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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

1718. **Application of statistics to the results of chemical analysis.** S. Mitsui and M. Arakawa (*Japan Analyst*, 1953, **2** [1], 6–13).—Errors of chemical analysis in routine work are statistically analysed and the results are discussed. Procedures that are not expressed or prescribed exactly are the main source of errors. Examples from the analyses of iron and steel are shown. K. SAITO

1719. **The application of perchloric acid in analytical chemistry.** E. Kahane (*Österr. ChemZtg.*, 1954, **55** [15–16], 209–215).—The uses of perchloric acid in analytical chemistry are reviewed. (84 references.) N. E.

1720. **Oxine and its derivatives. Some derivatives of 7-allyloxine of possible analytical application.** R. G. W. Hollingshead (*Research*, 1955, **8** [2], s9–s10).—The preparation of 7-allyl-5-nitroso-oxine (**I**) and 5-bromo-7-(2;3-dibromopropyl)oxine from 7-allyloxine (**II**) is described and their possible analytical use is outlined. With the use of **II**, pptn. occurs with Al, Cu, Zn, Ni, Ga, In and Mg (Mg in alkaline solution); no increase in selectivity is obtained with **I**, but sensitivity is increased. **I**, like 5-methyl-7-nitroso-oxine, gives a ppt. with Mg in acid solution and shows a marked increase in sensitivity towards Cu, for which the sensitivity limit in hot solution lies between 0·4 and 0·3 ml of 0·00001 *M* solution in a total volume of 7 ml. S.C.I. ABSTR.

1721. **The use of substituted dithiocarbamates in micro-analysis. III.** H. Malissa and E. Schöffmann (*Mikrochim. Acta*, 1955, [1], 187–202).—By introducing one of a number of substituents (N-acetylphenyl, phthaloyl or succinoyl) into the dithiocarbamate molecule the range of metal ions with which the compounds react could be varied, as were the sensitivity and the colours produced. The preparation of these carbamates is described in detail, as is that of ammonium tetramethylenedithiocarbamate. With this ammonium compound much smaller amounts of buffer are required to keep the pH constant than with the Na compounds. This reduction in total volume is of advantage. The selectivity of these reagents can be further improved by masking with cyanide. The greatest sensitivity was achieved by carrying out the spot test on a glass plate. Details of the applications of all four compounds are given. K. J. GARDNER

1722. **Analytical applications of Complexones. II. Qualitative analysis.** F. Bermejo Martínez and A. Prieto Bouza (*Inf. Quím. Anal.*, 1955, **9** [1], 10–21).—Modifications in the reactions of cations with inorganic and organic reagents in the presence of Complexones, the development of a general scheme for the qualitative analysis of cations, and the use of Complexones in the analysis of the alkaline earths and rare earths are reviewed. L. A. O'NEILL

1723. **Determination of specific surface of sieve-size powders.** B. Dubrow and M. Nieradka (*Anal. Chem.*, 1955, **27** [2], 302–305).—A simple air-permeability method for determining the specific surface or mean particle-diameter (D) of powders having D from 50 to 1000 μ is described. The apparatus includes a sensitive, adjustable flowmeter. After leaving the sample-tube the air is divided into two paths, *viz*, through a fritted-glass air-diffusion tube and into a water manometer; the manometer and the resistance formed by the diffusion tube together constitute a flowmeter. The apparatus is calibrated initially by determining the permeability const. of coarse, medium and fine glass diffusion-tubes. The values of D are calculated from the Gooden - Smith equation (*Brit. Abstr. B*, 1940, 768). The accuracy and precision of the method are adequate for routine analyses; one determination takes $\simeq$ 10 min. W. J. BAKER

1724. **Kinetics and thermodynamics of grinding processes. The use of radioactive isotopes.** G. F. Huttig, W. Simm and G. Glawitsch (*Monatsh. Chem.*, 1954, **85**, 1124–1132).—Sieve fractions of a glass powder were exchanged for corresponding fractions prepared from a glass containing ^{60}Co, and the distribution in further grinding operations of the unlabelled and labelled glasses was determined. The validity of a reaction of the first order is proved for the milling treatment, and from this and the distribution function the distribution coefficient and equations describing the grinding processes are derived. The use of the labelled fractions makes possible the discovery and evaluation of errors in the sieve analysis and reveals peculiarities before unknown. R. J. MAGEE

1725. **Measurement, control and continuous recording of the viscosity of fluids.** R. Fouché (*Chim. Anal.*, 1955, **37** [2], 56–57).—Viscosities up to 500,000 centipoises may be determined instantaneously on 2-ml samples of liquid under conditions of high temp. and pressure with apparatus using ultrasonic oscillations (28 kilocycles per sec.). The reproducibility is better than $\pm$ 2 per cent. and the accuracy better than $\pm$ 5 per cent. ($\pm$ 2 per cent. for viscosities $<$1000 centipoises). A. R. ROGERS

1726. **Practical method for estimating surface tensions of solutions.** M. Tamura, M. Kurata and H. Odani (*Bull. Chem. Soc. Japan*, 1955, **28** [1], 83–88).—A simple semi-empirical method for calculating the surface tensions of solutions is described; it is based upon the assumption of the additivity of the parachor (P) in the surface layer but not in the liquid bulk. The method is applicable to mixtures of liquids with widely differing polarities, for which the Meissner and Michael method cannot be used. The percentage error, $\{(\gamma_{calc} - \gamma_{obs})/\gamma_{obs}\} \times 100$, is within 10 per cent. when $P < 5$, and within 20 per cent. for large values of P. G. R. WHALLEY

1727. **The application of the morphology and optical properties of crystals in qualitative micro-analysis.** H. Jurány (*Mikrochim. Acta*, 1955, [1], 146–152).—It is considered that a knowledge of its crystal properties may often be of diagnostic help in the identification of a substance. This is illustrated by a number of examples. The omissions in the literature of these properties are pointed out. The author proposes the establishment of a codex of crystal forms and of a suitable terminology.

K. J. GARDNER

1728. **An economical analytical scheme for certain types of routine testing.** G. L. Buc (*Applied Spectroscopy*, 1954, **8** [4], 208).—A specification often stipulates a maximum limit (*L*), *e.g.*, 100 p.p.m., for a certain impurity. Where the average impurity in samples is well below this value, *e.g.*, 10 p.p.m., it may be possible to halve the number of determinations by analysing in pools of two so that the acceptable limit is 50 p.p.m. Pools of greater than two (*n*) may also be practicable with a correspondingly lower acceptance limit $\frac{L}{n}$. When the pool analysis exceeds the acceptance limit, it must be broken down into smaller components. The principle can be applied to both upper and lower limits, in all cases where it is possible to pool samples, and where the property being investigated is additive.

P. T. BEALE

1729. **Improvements in or relating to titration method and apparatus.** H. A. Frediani (Brit. Pat. 719,995, Date Appl. 7.11.52).—Automatic titration is effected in cases exemplified by the Karl Fischer titration, by timing the temporary excursions to end-point conditions caused by slowness of the mixing, extraction of water from the experimental material, etc. The titration is stopped when the duration of this end-point condition reaches a preset value. A circuit is described for completely automatic titration by the dead-stop technique. The depolarisation current actuates the timer, which is stopped and reset when the current falls again to the polarisation value. The method can also be used for the back-titration.

E. J. H. BIRCH

1730. **A fundamental research with the aid of the high-frequency titrimeter of resistant type. (The relation between the resistance and the concentration of the solution and its application to the explanation of titration curves.)** K. Nakano (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [8], 773–776).—The resistance - concn. diagram of a no. of electrolytes obtained by the use of the high-frequency titrimeter ($\simeq$ 1 Mc.p.s.) of resistant type (K. Nakano *et al.*, *Anal. Abstr.*, 1954, **1**, 2031) is closely studied in order to elucidate the mechanism of titrations. For strong electrolytes a max. resistance is always observed at a concn. ($\simeq 10^{-3}M$) which is proportional to the frequency. Weak as well as strong acids show the same pH value ($\simeq$ 3·85) at the concn. of max. resistance. Curves for weak acids can be regarded in the same way as those for strong acids, when the degree of dissociation is taken into account. The law of additive properties roughly holds for mixtures of strong electrolytes, so that the neutralisation titration curve can be predicted from the resistance - concn. diagram of the reactant and the product; this accords with the experimental results. The titration curve involving a weak acid or base can also be accounted for by taking the degree of dissociation into consideration.

K. SAITO

1731. **A fundamental research on high-frequency titrations. [Theoretical considerations.]** K. Nakano (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [8], 776).—Since the resistance - concn. diagram is fundamental for high-frequency titration (*Anal. Abstr.*, 1955, **2**, 1730), it is essential to make clear the basis of the curve. By introducing an equivalent circuit, which represents both the inductance and capacitance of the circuit, the diagram is clearly interpreted. Essentially, high-frequency titration is a modified conductimetric titration. The max. on the diagram appears to be due to the capacitance of the soln. (including the vessel). It can be extinguished by electrostatically shielding the vessel, *e.g.*, by sticking aluminium foil on either the inside or outside of the glass vessel. Thus titrations at an intermediate concn. ($\simeq 10^{-3}$ *M*) of the soln., which otherwise cannot be done, are made possible.

K. SAITO

1732. **Titration of acids in dimethylformamide using high frequency.** J. A. Dean and C. Cain, jun. (*Anal. Chem.*, 1955, **27** [2], 212–214).—A high-frequency oscillator was applied to the titration of acids in dimethylformamide solution. Sharp V-shaped titration curves are obtained for strong acids and NH_4^+ over the range of the sensitivity of the instrument and include concn. as low as 0·0001 *M*. Less acute angles are obtained for acids of intermediate strength and, in general, adequate end-point determination is possible for all acids whose pK_a values do not exceed 7 in water.

G. P. COOK

1733. **The automatic analysis of gases.** D. Saint-James (*Chim. Anal.*, 1955, **37** [2], 31–38).—This is a review of methods for the continuous automatic analysis of gases, by the use of solubility in liquid absorbent, electrical conductance of solution in electrolyte, thermal conductance, paramagnetism, or i.r. absorption.

A. R. ROGERS

1734. **Theory and practice of chromatography.** R. A. Wells (*Pharm. J.*, 1955, **174**, 25–26; 59–60).—A simple exposition is given of the theory of frontal analysis, displacement development and elution development of chromatograms, and comments are made on the principles and application of partition chromatography.

S.C.I. ABSTR.

1735. **Quantitative paper chromatography with the photo-electric "leukometer." The use of paper chromatography in nutritional chemistry problems.** H. Sulser (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [6], 518–527).—A new method is described for the quantitative evaluation of paper chromatograms with the help of the "leukometer" (B. Lange, Berlin). A multiflex galvanometer serves for the registration of the small photo-current. The sensitivity of this galvanometer can be varied. One reading per spot is sufficient for a determination and there is no need to make the paper transparent, since reflected light is used. Graphic representation gives, for the points of a series and within a certain concentration range, a linear relation between concentration and the log. of the galvanometer deviation. The apparatus can be used, also, for the measurement of the intensity of whiteness or colour of solids or powders, as well as for coloured solutions.

R. J. MAGEE

1736. **Glass-fibre paper impregnated with silicic acid as a new chromatographic tool.** J. W. Dieckert and R. Reiser (*Science*, 1954, **120**, 678).—Mono-, di- and tri-palmitin, cholesterol and cholesteryl acetate can be separated on glass paper impregnated with

silicic acid with the use of a developing solvent comprising 2 per cent. of diethyl ether in *iso*octane. Location of the sterol spots was made by spraying one side of the chromatogram with Liebermann - Burchard reagent, followed by heating over a hot-plate with exposed heating element; the cholesterol and its acetate appear as bright pinkish-red spots. The reverse side was then sprayed with a dichromate - H_2SO_4 - H_2O soln. followed by heating over the hot-plate, when all areas containing carbon compounds appeared as light to dark grey - black spots on a yellow - orange background.

H. F. W. KIRKPATRICK

1737. **Recent developments in the technique of electrophoresis.** E. Wiedemann (*Int. Arch. Allergy*, 1954, **5** [1], 1–22).—A review is presented. Certain advances in technique are considered and a number of interferometric methods of recording are described. D. F. HAWKINS

1738. **Potential errors in spectrophotometry with optically dense solutions.** A. H. Mehler (*Science*, 1954, **120** [3129], 1043–1044).—The limitations of optical instruments are emphasised with regard to the measurement of optical densities in soln. of high background density. Valid measurements of high optical densities are attainable only if the light used is monochromatic. H. F. W. KIRKPATRICK

1739. **Some observations on sparked craters in spectrographic micro-volume analysis.** J. K. Hurwitz (*Spectrochim. Acta*, 1955, **7** [1], 1–13).—The effective spark distribution in a point-to-plane discharge was determined by making depth measurements on sparked craters produced in Mg, Zn and Cd. The distribution function is approx. a paraboloid of revolution having a mean slope of 1·68. For Mg, the rate (R) of bulk volatilisation is independent of traverse speed (s), although the radius (r) and depth (z) of the crater vary with s; equations relating r, z and s are deduced. Extension of the measurements to sparked craters of other metals and alloys indicate that R tends to increase as the m.p. and thermal conductivity decrease. The sensitivity limits in the spectrographic analysis of solids depend also on values of R. Application of these data to micro-volume analysis is discussed. W. J. BAKER

1740. **Quantitative spectrochemical analysis by the constant-temperature arc method.** N. W. H. Addink, with J. A. M. Dikhoff, C. Schipper, A. Witmer and T. Groot (*Spectrochim. Acta*, 1955, **7** [1], 45–59).—The constant-temperature arc method of quant. spectrochemical analysis, as developed by Addink, is described fully. The method consists in volatilising completely 5 mg of a powdered sample in the shallow anodic crater of a carbon arc, with the addition of materials (*e.g.*, SiO_2, C, $BaCO_3$, Ni) to modify the rate of volatilisation. The line intensities are calibrated by comparison with selected Fe lines, and the percentage concn. of any element in the sample is given by the product of K and I, where K = proportionality factor and I = line intensity. Values of K, as determined experimentally for 235 spectral lines of 38 elements, are reported, together with some results of applying the method to the determination of Na in glass and of Si, Mo, Al and Fe in tungsten. W. J. BAKER

1741. **Effective line widths in spectrophotometry.** J. Junkes and E. W. Salpeter (*Spectrochim. Acta*, 1955, **7** [1], 60–63).—The use of line widths in quant. spectrochemical analysis is discussed, particularly with reference to recently developed photometers for measuring the effective diameters of photographic star images. Photographic widths of spectral lines are as accurate as densities, besides covering a much wider range of intensities. Some data for Fe lines used as intensity standards are reported, and the need for a "variable-slit photometer" for measuring effective widths of spectral lines is indicated. W. J. BAKER

1742. **Quantitative analysis of powders by means of infra-red absorption spectra. I. Theory.** G. Duyckaerts (*Spectrochim. Acta*, 1955, **7** [1], 25–31).—A complete theoretical treatment confirms the validity of the proposed formula representing the experimental data (*Anal. Abstr.*, 1954, **I**, 2296) for the influence, on i.r. absorption, of the particle size of a powder dispersed in a KBr disc. The extinction ($E = \log I_o/I$) for a dispersion of uniform cubical particles sufficient to provide n layers in a transparent medium can be represented by

$$-(m/K\rho sd) \log [(1 - K) + Ke^{-kd}]$$

where m = mass of particle, d = cube edge, ρ = sp. gr., s = area of disc normal to the beam, K = fraction of surface covered by a layer, and k = mean coeff. of intrinsic absorption. For spherical particles this value of E is multiplied by 1·5. When kd is $< 0{\cdot}3$ the dispersion is quasi-mol. and the measured extinctions follow the Beer - Lambert law. Experiments indicate that the absorption curve for a dispersion of non-uniform particles, when expressed in terms of the diameter of the max. of the grading curve, simulates the absorption curve for a dispersion of uniform-sized particles. It is not necessary, in practice, to obtain the grading curve for each sample, provided that it is known to what degree of fineness the sample should be ground to ensure an approx. mol. dispersion in the disc of KBr. W. J. BAKER

1743. **Quantitative analysis of powders by means of infra-red absorption spectra. II. Experimental.** J. Bonhomme (*Spectrochim. Acta*, 1955, **7** [1], 32–44).—The effect of particle size on the intensity of the i.r. absorption bands of calcite (710, 876 and 1431 cm^{-1}) and of Dowex-50 synthetic resin (1001 and 1028 cm^{-1}) was determined on samples carefully graded by sedimentation, each fraction (varying from 0·001 to 26·5 μ) being mixed with finely powdered KBr and moulded into discs. The results are reported and discussed in relation to the theory developed in Part I (*cf. Anal. Abstr.*, 1955, **2**, 1742). In general, they indicate that the absorption by a powder of uniform- or non-uniform-sized particles follows the same law, except that K and k depend on the grading curve. The values of E are const. to within $\simeq$ 3 per cent. for all values of $kd < 0{\cdot}3$. W. J. BAKER

1744. **Calcium carbonate as an internal standard for quantitative infra-red analysis.** L. E. Kuentzel (*Anal. Chem.*, 1955, **27** [2], 301).—Pure $CaCO_3$ is a useful internal calibration standard for the i.r. absorption analysis of solids; it is stable, inert, has three widely spaced sharp bands and can be prepared with all particles $< 1\mu$ in diameter. Its use (1 per cent. by wt. of sample) in calibrating mixtures of sodium toluenesulphonate and alkylbenzenesulphonate (40 to 60 per cent. of each in the other) is described in detail. The calibration was effected in mineral-oil suspensions by the method of Barnes *et al.* (*Brit. Abstr. C*, 1948, 64). The ratio of the absorbance at the 11·4-μ band of $CaCO_3$ to that at a characteristic band for each of the other two

compounds is plotted against the concn. of the corresponding compound in a synthetic mixture, so as to obtain two linear calibration curves.
W. J. BAKER

1745. **Use of multi-channel recording in X-ray fluorescent analysis.** M. F. Hasler and J. W. Kemp (*A.S.T.M. Symp. on Fluorescent X-Ray Spectrogr. Anal.*, 1954, 34–42).—The application of a multi-channel ratio system to X-ray fluorescent analyses, particularly a non-dispersive analyser, was investigated. Ratios of the analysing channel to a standard channel were obtained, the channels being provided with suitable filters to produce max. radiation of given elements in individual channels. Application of the method to the analysis of a multicomponent light-metal alloy is described, showing the method of correction for the presence of other elements on individual determinations. The ratio system shows an improved accuracy compared with single-channel, absolute determinations, for which the source, power supply, etc., are not constant. METALL. ABSTR.

1746. **Analytical applications of the Barker square-wave polarograph.** D. J. Ferrett and G. W. C. Milner (*Analyst*, 1955, **80**, 132–140).—Some advantages of the square-wave polarograph and its application to a number of analytical problems are discussed. It is specially suitable for the analysis of alloys, *e.g.*, the direct determination of Cu, Pb, Cd, In and Zn in alloys, without previous chemical separation. The determination of these elements in Zn-, Mg- and Sn-based alloys is described. Other applications include the determination of impurities (Cu and Pb) in analytical reagents. In *M* H_2SO_4, the peak for U is clearly separated from both the peaks for Pb and Cu.
A. O. JONES

1747. **Simplified procedure for computing the growth of radioactive decay products.** H. W. Kirby and D. A. Kremer (*Anal. Chem.*, 1955, **27** [2], 298–299).—A method, based on the modified Bateman equation, for facilitating the calculation of growth of decay products from radioactive materials, especially when the decay chain is long, is presented. A table of functions for the Th series is given and its use for calculating the equation of growth of ^{220}Rn from ^{232}Th is explained. W. J. BAKER

See also Abstract 2012.

2.—INORGANIC ANALYSIS

1748. **New techniques for analysing mixtures of trace metals.** R. M. Sherwood and F. W. Chapman, jun. (*Anal. Chem.*, 1955, **27** [1], 88–93).—Procedures for the unequivocal determination of V, Fe, Ni, Mn, Ti, Pb, Cu, Cr and Pt, especially in used petroleum catalysts and petroleum residues, are critically examined and the following recommended procedures are described. The catalyst sample (10 g) is ground and ignited at 550° C for 1 hr.; from this, 1 g is dissolved in conc. H_2SO_4 (2 ml) and HF (5 ml), excess of HF being removed by fuming, and the cooled residue is dissolved in water (5 to 10 ml). After removal of organic materials from petroleum residues the inorganic residue is treated similarly. The various elements in the resulting solutions are analysed as follows.—*Vanadium.* Interfering elements are removed over a mercury cathode. The V in the soln. is converted into a tungstovanadophosphoric acid which is extracted with *iso*butanol and the V is determined spectrophotometrically at 400 mμ. *Iron.* The reagent 4:7-diphenyl-1:10-phenanthroline (bathophenanthroline) (Smith, McCurdy and Diehl, *Brit. Abstr. C*, 1952, 567) is recommended for the colorimetric determination of Fe. *Nickel.* Extraction from 0·3 *N* aq. NH_3 with a 0·006 per cent. dithizone soln. in $CHCl_3$, dilution with $CHCl_3$ and measurement of the absorption at 665 mμ is selected. *Manganese.* Oxidation to MnO_4' is carried out with periodate in a phosphoric acid medium, after previous removal of Fe, Cu, etc., by extraction of the cupferrates with $CHCl_3$. The MnO_4' soln. is made just alkaline and the absorption measured at 525 mμ. This avoids interference by Cr. *Lead.* Copper is removed by extraction with dithizone in CCl_4 in acid solution. The Pb is then extracted with dithizone in $CHCl_3$ from an ammoniacal solution. Bismuth, if > 10 μg per ml, interferes. *Chromium.* A soln. of the sample (60 to 700 μg) is electrolysed as in the determination of V. The Hg is distilled, the residue being dissolved in dil. H_2SO_4 and made alkaline with *N* NaOH; the solution is heated with Br for 5 min., made acid with H_2SO_4 and boiled to expel Br. Phosphoric acid is added and the absorption measured at 440 mμ. *Platinum.* The coloured complex with $SnCl_2$ is formed after reduction of all the Pt to the metal and dissolving in aqua regia, evaporating to a moist residue and re-dissolving in water. The absorption is measured at 400 mμ.
D. A. PANTONY

1749. **Use of chromatography in the detection of heavy metals.** A. G. Kniga and V. I. Ustinskaya (*Tr. Leningr. Tekhnol. In-ta Pischchevoĭ Prom-sti*, 1953, **3**, 154–159; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 39,989).—The following combinations of heavy metals are separated on an alumina column: (with Na_2S soln. as developer) Cu and Cd; Cd and Pb; Cu and Ni; Zn, Cd and Fe^{III}; Sb^{III}, Cd and Zn; (with NaOH soln. as developer) Fe and Ni; Fe and Co; Fe and Cu; Fe, Ni and Co; Fe^{II} and Fe^{III}; Hg^{I} and Hg^{II}; Cu^{I} and Cu^{II}; Mn^{VI} and Mn^{VII}. Mn^{II} must be oxidised with NaOBr or bromine water. The methods are suitable for the detection of small amounts of impurities in technical reagents and in minerals. E. HAYES

1750. **Systematic investigations on the applicability of diethyldithiocarbamate in analysis. IV. Formation of metal diethyldithiocarbamates and their extractability in relation to the pH of the solution.** H. Bode (*Z. anal. Chem.*, 1955, **144** [3], 165–186).—The composition of various metal dithiocarbamates, and conditions of their pptn. and extraction at different pH values, are presented. Absorption spectra of their solutions in CCl_4 are recorded. Effects of other complexing agents present, *e.g.*, KCN and Complexone III, are summarised. Applications to the analysis for Cu^{II}, Ag, Au^{III}, Zn, Cd, Hg^{II}, In, Tl^{I}, Tl^{III}, Sn^{IV}, Pb^{II}, As^{III}, Sb^{III}, Bi, V^{V}, Se^{IV}, Te^{IV}, U^{VI}, Mn^{II}, Fe^{III}, Ni^{II}, Co^{II}, Pd^{II} and Pt^{IV} are described. The reagent is not suitable for alkali and alkaline-earth metals, Al, Sc, Y, La, Ga, Ge, Ti^{IV}, Zr^{IV}, Th^{IV}, Nb^{V}, Ta^{V}, Cr^{VI}, Mo^{VI}, W^{VI}, Ru, Rh, Os and Ir. D. R. GLASSON

1751. **Spectrochemical determination of metallic elements in non-metallic samples.** J. M. Gillette, B. R. Boyd and A. A. Shurkus (*Applied Spectroscopy*, 1954, **8** [4], 162–168).—The paper outlines suitable methods for several different problems. Wear products are determined in lubricating oils by the rotating disc electrode method, and coefficients of variation of $\simeq 5$ per cent. of the content

are claimed for Si, Pb, Ca, Fe, Cu, Cr and B. Cements are analysed by mixing with anhydrous B_2O_3 and graphite and pressing into the form of a rotating disc electrode. Preliminary furnace fusion with $LiCO_3$ - B_2O_3 mixture is recommended when working with widely varying minerals and oxides, and typical results are given for nickel ores. Sparking on to the molten surface of this type of sample should remove effects due to variations in physical form. X-ray fluorescence is applied to such materials as silica gel and a table of estimated limits of detection in different matrices is given for this method. P. T. BEALE

1752. **Relations between the segregation of elements and sampling methods of metal blocks for chemical analysis. I.** J. Kashima, K. Yasuda and K. Sanada (*Japan Analyst*, 1953, **2** [2], 108–112).—The segregation of minor constituents in metals is studied with the aid of the spark spectrographic method. A high-voltage spark (8 kV, 0·004 μF, 0·0083 mH) is struck between a pure silver rod and the flat section of the metal to be examined, the gap being 0·3 to 0·4 mm. The reproducibility of the analytical result is carefully examined. The segregation of Si (1·96 per cent.) and Mn (0·59 per cent.) in cast iron, and Cu (3·8 per cent.) in Al is examined on cross, vertical and diagonal sections. The segregation depends on the thickness and the rate of cooling of the metal sample. The silicon contents are generally high in the periphery (2·0 to 2·4 per cent.) and in the centre ($\simeq$ 2·0 per cent.), and low in the intermediate part (1·6 to 1·9 per cent.). Mn in Fe and Cu in Al do not show this tendency, but marked local segregations are observed at random positions. The homogeneity is greater the higher the rate of cooling. Segregation of certain components is generally observed; this requires a more precise study of the relationship between the structure and the chemical composition of the metal. K. SAITO

1753. **The quantitative spectrochemical estimation of traces of metals [in metals and alloys].** F. Thümmler and I. Morgenstern (*Chem. Tech.*, 1955, **7** [1], 10–16).—The quantitative spectroscopic methods for estimating traces of metal impurities in metals and alloys are discussed and an improved method is described in detail. In this method, the comparative arc spectra of the sample and of the pure metal, dosed with ranges of known additions of the impurity, are taken by arcing the sample and comparative pieces in the form of pellets of the oxides mixed with graphite or powdered Ag or Cu. To prepare the sample pellet, 0·2 g of the metal to be tested is dissolved in pure HNO_3 and the resulting nitrate, after ignition to oxide, is mixed with powdered graphite (2 pt. of oxide to 5 pt. of graphite), and the mixture is pressed under $\simeq$ 50 kg per sq. mm into pellets of 6 mm diameter and 2 to 3 mm in height. The comparison pellets are made in a similar manner from mixtures in definite proportions of a standard solution (2 per cent.) of the nitrate of the pure metal and of a (0·02 per cent.) solution of the nitrate of the impurity, the salts being ignited to oxide before mixing with the graphite. Typical analyses of Cd in Zn, Pb in Zn, Ni in Fe, Pb in Ni, Cr in Fe, and Cd in Ni are described. H. L. WHITEHEAD

1754. **A simplified procedure for the determination of index point and index line in the step-filter technique of spectrographic analysis.** K. V. Chinnappa (*Trans. Indian Inst. Metals*, 1952, **6**, 339–341 [published 1954]).—The mean value of $\Delta D/\Delta Dm$ is plotted against the log. of the concn. in per cent. for each of the standard samples, where ΔD is the d (optical density) difference between the analysis line and the internal standard and ΔDm is the d difference between two steps of the step-filter. By simple algebra $\left(\frac{\overline{\Delta D}}{\Delta Dm}\right) = \frac{\Sigma_1^n \Delta D}{\Sigma_1^n \Delta Dm}$. From the resulting straight line the index point and the index line can easily be read. METALL. ABSTR.

1755. **A method for the spectrographic analysis of alloys having special compositions.** K. V. Chinnappa (*Trans. Indian Inst. Metals*, 1952, **6**, 342–343 [published 1954]).—To offset the increased plate error due to the large intensity ratio, a reference sample (an accurately analysed sample of the special type) is used as a standard. The mean value of $\frac{\Delta D}{\Delta Dm}$ for the reference sample is determined, where ΔD is the d (optical density) difference between the analysis line and the internal standard and ΔDm is the d difference between two steps of the step-filter, and a modified calibration curve is made by drawing a line through this point parallel to the calibration curve of $\left(\frac{\overline{\Delta D}}{\Delta Dm}\right)$ plotted against the log. of the percentage composition for the standard samples. METALL. ABSTR.

1756. **Mechanism of the Karl Fischer titration and a new modification of this method.** E. Bonauguri and G. Seniga (*Z. anal. Chem.*, 1955, **144** [3], 161–164).—The influence of methanol on the Karl Fischer titration is investigated. The deviations found vary with the age of the reagent. In the presence of a large excess of SO_2, the reaction $I_2 + SO_2 + 2H_2O = H_2SO_4 + 2HI$ is favoured, especially with larger amounts of H_2O. The intermediate product, $C_5H_5N{\cdot}SO_3$, has been prepared pure and reacts with both water and methanol. Errors are minimised and calculable when methanol diluted with dioxan and a reagent containing iodine, sulphur dioxide, pyridine and methanol (1:2:9:16) are used; as little as 1 mg of H_2O per ml can be determined. D. R. GLASSON

1757. **Alkali metals in phosphating and cyanide-plating baths. Determination by means of anion exchangers.** G. Gabrielson (*Metal Finish.*, 1955, **53** [2], 58–60).—The sample solution (5 ml) (zinc cyanide baths are diluted 5 times) was passed through a layer of a strongly basic anion-exchange resin in the hydroxyl form. The resin was then washed with distilled H_2O (50 ml). Flow rates during filtration and washing were 3 to 4 ml per min. The combined eluate and washings were titrated with 0·1 N HCl, methyl orange being used as indicator. The eluate contains only alkali hydroxide. The maximum relative error was $< 0{\cdot}5$ per cent. W. ASHWORTH

1758. **Correction procedure for the flame-photometric determination of sodium in the presence of calcium.** F. Hegemann and B. Pfab (*Glastechn. Ber.*, 1954, **27** [6], 189–192).—The presence of calcium causes errors in the flame-photometric determination of sodium. These errors are dependent on the Na and Ca concentration and on the total concentration of the solution. Correction procedures are applicable only within a narrow range and depend in principle on interpolation between Na

and Ca intensity - concentration curves with constant Ca and Na additions. For the complete elimination of the Ca influence, this element must be chemically removed. A correction procedure is described for the accurate flame-photometric determination of Na in lime - soda glass by the use of a reference solution. Its range of application lies between 0·023 and 0·027 per cent. of Na in the solution. The accuracy is ± 0·5 per cent. of the Na_2O content. N. E.

1759. **Application of chloroplumbic acid to direct detection of potassium ion.** W. Rodziewicz and J. Szychliński (*Roczn. Chem.*, 1954, **28** [2], 657–661).—Solutions of H_2PbCl_6 may be prepared by saturating suspensions of $PbCl_2$ in conc. HCl with Cl at ⩾ 10° C, by adding lead tetra-acetate to conc. HCl at 0° C, or by oxidising suspensions of $PbCl_2$ in conc. HCl with 25 per cent. H_2O_2, $KClO_3$, $NaBO_4$ or MnO_2; the first method is preferred. The solutions may be stored for several months without decomp. at ⩾ 10° C. The solution, 10 ml containing ⩽ 5 mg of K, is evaporated down to ≃ 1 ml and 1 ml each of conc. HNO_3 and HCl are added; the mixture is then ignited to eliminate ammonium salts. The residue is extracted with 1 ml of warm 10 per cent. HCl, filtered and 0·5 ml of conc. HCl is added to the filtrate, which is then cooled to − 10° C, and 2 ml of H_2PbCl_6 solution at − 12° C are added. A yellow ppt. of K_2PbCl_6 is seen after a few min. in the presence of ⩽ 3 mg of K per ml. The reaction cannot be applied in the presence of Rb and Cs; Hg, Bi, Cu, Cd, As, Sn, Sb, Fe, Cr, Al, Mn, Zn, Ni, Co, Ba, Sr, Ca, Mg, Li and Na do not interfere. R. Truscoe

1760. **Sodium tetraphenylboron for potassium detection in systematic qualitative procedures.** R. F. Muraca, H. E. Collier, J. P. Bonsack and E. S. Jacobs (*Chemist Analyst*, 1954, **43** [4], 102–103).—A procedure is described for the complete removal of ammonium salts before the use of sodium tetraphenylboron for the detection of potassium. G. B. Thackray

1761. **Indirect polarometric (amperometric) determination of potassium, using sodium tetraphenylboron.** W. Kemula and J. Kornacki (*Roczn. Chem.*, 1954, **28** [4], 635–641).—The solution, containing ⩽ 1 mg of K in concn. ⩽ 0·0005 *M*, is adjusted to pH 5 to 6 by adding 50 per cent. acetic acid, and *N* $NaNO_3$ is added to approx. 0·1 *N*, followed by 0·002 *M* $NaB(C_6H_5)_4$, to ppt. $KB(C_6H_5)_4$. Gelatin is added, to a concn. of 0·01 per cent., and excess of $NaB(C_6H_5)_4$ is titrated polarographically with 0·005 *M* $TlNO_3$. The method cannot be applied in the presence of NH_4^+, Rb, Cs or Cl'. R. Truscoe

1762. **Turbidimetric micro-determination of potassium with sodium tetraphenylboron.** J. de la Rubia Pacheco and F. Blasco López-Rubio (*Inf. Quim. Anal.*, 1955, **9** [1], 1–9, 21).—One ml of solution containing 5 to 300 μg of K is placed in a colorimeter tube, evaporated at 70° to 80° C and allowed to cool; the residue is dissolved in 0·1 to 0·2 ml of a citrate - HCl buffer of pH 2. The Na tetraphenylboron reagent (0·2 g) is added, the mixture is shaken for 1 to 2 min., 1 ml of the buffer solution is added and H_2O to a vol. of 5 ml. The suspension is homogenised and the turbidity measured in a photometer. The reagent may be prepared by dissolving 3 g of the Na tetraphenylboron in 20 ml of either a borate - HCl buffer (pH 9) or a phosphate - citric acid buffer (pH 8), adding H_2O to a vol. of 100 ml, heating for 1 min. at 70° C and filtering, and is stable for ⩽ 30 days. The turbidities obey Beer's law within the range 5 to 100 μg of K in 5 ml of final dilution, but the method is still applicable at higher concn. Ca, Mg, Al, Fe^{++}, Fe^{+++}, Co, Ni, Mn, Cu^{++}, SO_4'' and PO_4''' do not interfere, but NH_4^+, Rb and Cs must first be eliminated. The method may be used for determining the available or exchangeable K_2O in soils. L. A. O'Neill

1763. **Electrophotometric determination of copper with phenylthiosemicarbazide.** S. Komatu and H. Utiyama (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [12], 1280–1283).—Phenylthiosemicarbazide (**I**) reacts with Cu^{++} in acidic soln. and gives an intense blue colour (Ruhemann, *J. Amer. Chem. Soc.*, 1931, **53**, 552). This reaction is utilised for the electrophotometric determination of Cu^{++}. The colour of **I** is suppressed in an acetic acid soln. > 1·7 *N*. The max. absorption of the Cu - **I** complex is at 650 mμ and the colour becomes stable 3 hr. after the addition of the reagent. The extinction coeff. of the complex soln. is proportional to the concn. of Cu^{++}(0·5 to 15 μg of Cu per ml) in 3 *N* acetic soln. Fe and Pt react with **I** under the same conditions and vitiate the determination. Ag and Hg (< 72 μg per ml) react with **I**, but do not interfere with the light absorption of the Cu complex. K. Saito

1764. **Complexometric titrations (chelatometry). X. Catechol violet, a new specific indicator: determination of copper.** V. Suk, M. Malát and A. Jeníčková (*Coll. Czech. Chem. Comm.*, 1955, **20** [1], 158–161).—Copper (< 20 mg in 100 ml) may be titrated directly in acid (Na acetate buffer) or alkaline (aq. NH_3 soln. - NH_4Cl buffer) soln. with disodium ethylenediaminetetra-acetate, with a 0·1 per cent. aq. soln. (4 to 5 drops) of catechol violet (catecholsulphonephthalein) as indicator. The colour changes at the end-point are, respectively, blue to yellow and blue to violet. The blue colour of the Cu complex interferes in stronger soln. Salts of Ag, Mg, Ca, Ba, Sr, ammonium or the alkali metals do not interfere with the titration in acetate buffer. [This is a translation into German of a paper originally published in *Chem. Listy*, 1954, **48**, 1511.] A. R. Rogers

1765. **New gravimetric method for the determination of silver.** P. Spacu and M. Hlevca (*Comun. Acad. R.P. Române*, 1953, **3** [5–6], 211–215; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 40,008).—Potassium xanthate precipitates Ag quant. as $C_2H_5OCSSAg$, which is insoluble in water, ethanol and ether. Potassium xanthate soln. (15 per cent.) and 1 to 2 drops of aq. pyridine soln. (1 + 3) are added to a slightly acid aq. soln. containing Ag. The ppt. is collected on a sintered-glass crucible, then washed with water, ethanol and ether and, finally, dried for 20 min. in a vacuum-desiccator. E. Hayes

1766. **Quantitative analysis without separation. XIII. Gravimetric analysis without separation for the systems Ag - Pd and Ag - Hg (chlorides).** N. Unohara (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [12], 1275–1277).—Thermal decomposition curves of PdI_2, AgI - PdI_2, AgCl, $HgCl_2$ and AgCl - $HgCl_2$ are examined with a thermobalance, and their application to gravimetric analysis is discussed. PdI_2 behaves in a complex way on being heated and the determination of Pd in Ag - Pd mixtures is not satisfactory. The amount of Hg (> 30 mg in

600 mg of the mixture) in the mixed ppt. of AgCl and Hg_2Cl_2 can be calculated from the difference in loss of weight on heating at 100° and 590° C.

K. SAITO

1767. **A new sensitive method for the detection of beryllium.** M. Theis (*Z. anal. Chem.*, 1955, **144** [3], 192–194).—By the use of Chromazurol S in weakly acid media, pH $\simeq$ 6, Be can be detected to a sensitivity of 1 in 4,500,000 on a dropping-plate. Detection in the presence of Al and alkaline-earth metals is described. Interference by Fe^{III}, Zr and Cu is avoided by masking with tartaric acid, NaF and Complexone III, respectively.

D. R. GLASSON

1768. **Fluorescence detection and determination of beryllium with 2-o-hydroxyphenylbenzothiazole.** Z. Holzbecher (*Coll. Czech. Chem. Comm.*, 1955, **20** [1], 193–197).—Salts of Be fluoresce blue with 2-*o*-hydroxyphenylbenzothiazole (**I**) in aq. ethanolic soln. at pH 4·5 to 5. The limit of detection is 0·05 p.p.m. The sensitivity is reduced in the presence of Fe^{III}, Cr, Al, Bi, Sb, Sn^{IV}, Zr, Ti, Cu^{II}, Zn, fluoride or arsenate. By using a calibration curve, 1 to 30 μg of Be in 5 ml of soln. may be determined quant. by mixing with acetate buffer of pH 5 (10 ml), 0·01 *M* **I** in ethanol (1 ml) and ethanol (to 50 ml), and comparing the fluorescence with a quinine sulphate standard. [This is a translation into German of a paper originally published in *Chem. Listy*, 1954, **48**, 1156.] A. R. ROGERS

1769. **A new organic reagent for the determination of microgram quantities of beryllium by a spectrophotometric method.** J. H. Wood (*Mikrochim. Acta*, 1955, [1], 11–24).—The method described is suitable for the quant. determination of 0·2 to 1·6 μg of Be per ml and was devised for the determination of Be in contaminated air. It depends on the spectrophotometric measurement of the coloured complex formed between Be and the sodium salt of the dye Solochrome Brilliant Blue B (**I**) (Colour Index No. 723). The soln. under test (1 vol.), 0·2 per cent. w/v of **I** in water (2 vol.) and 5 per cent. aq. ammonium acetate soln., pH 7 (2 vol.), are measured into a 1-cm quartz cell of the Unicam quartz spectrophotometer. The absorption is measured at 550 mμ after 30 min. The strength of absorption falls off only slightly after 24 hr. It is advisable to make a new standard curve for each supply of dye. K. J. GARDNER

1770. **The spectrochemical estimation of magnesium in spherulitic cast iron.** F. Thümmler and I. Morgenstern (*Chem. Tech.*, 1955, **7** [1], 35).—In the spectrochemical estimation of Mg in spherulitic cast iron, the pellets (see *Anal. Abstr.*, 1955, **2**, 1753) must be made by mixing the oxides with powdered Ag, because of the magnesium content of graphite. The estimation of Mg in six sample pellets gave results ranging from 0·067 to 0·077 per cent.

H. L. WHITEHEAD

1771. **Precipitation of calcium as calcium oxalate in weakly acid media.** W. A. C. Campen (*Chem. Weekbl.*, 1955, **51** [10], 188).—A procedure for obtaining a satisfactory pptn. of calcium oxalate from weakly acid media is described. The solution is made slightly acid with HCl. Ammonium oxalate is added and the liquid is boiled. HCl is added until any ppt. formed disappears. Ammonium acetate soln. (15 per cent.) is added slowly and with stirring until the liquid is just alkaline to bromophenol blue. Aq. NH_3 is then added, followed by more ammonium acetate, and the mixture is heated for 1 hr. on the water bath.

A. J. MEE

1772. **Rapid removal of iron, aluminium, titanium, manganese and phosphoric acid from solutions in which magnesium and calcium are to be determined.** W. A. C. Campen and L. J. H. Nijst (*Chem. Weekbl.*, 1955, **51** [10], 186–188).—The ammonium benzoate method for the determination of Mg and Ca has been improved so that Fe, Ti, Al, Mn and PO_4''' are rapidly removed from the solution before the precipitation. For every 25 mg of P_2O_5 present, 1 ml of a 10 per cent. solution of $FeCl_3$ is added, and for every 5 mg of Mn, 1 ml of 1 per cent. $KMnO_4$. The weakly acid solution is boiled for 2 min., when PO_4''' is precipitated as $FePO_4$ and Mn^{II} as MnO_2. The excess of $KMnO_4$ is removed by boiling with ammonium benzoate at a pH of about 4. The method described by Kolthoff *et al.* (*J. Amer. Chem. Soc.*, 1934, **56**, 812) is then carried out. A. J. MEE

1773. **Polarographic determination of zinc.** I. A. Korshunov (*Uch. Zap. Gor'kovskogo Un-ta*, 1953, [24], 15–20; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 41,694).—The conditions for the reduction of zinc in solutions of various indifferent electrolytes are described. Methods are given for the polarographic determination of zinc in metallic cadmium, magnesium and aluminium alloys, lead, ores, galvanic-bath electrolytes and air. E. HAYES

1774. **Co-precipitation of zinc bismuthithiocyanate and mercurithiocyanate.** L. Boguslawski and A. Cygański (*Roczn. Chem.*, 1954, **28** [2], 667–668).—A brick-red ppt. appears when $<$ 0·0075 *M* solutions of zinc salts are added to a mixture of 5 ml of 0·5 *M* $Bi(NO_3)_3$, 2 ml of 2 *M* NH_4CNS and 1 ml of 0·2 *M* $Hg(NO_3)_2$. The ppt. consists of 1 : 12 to 1 : 67 mixtures of $Zn_3[Bi(CNS)_6]_2$ and $ZnHg(CNS)_4$.

R. TRUSCOE

1775. **5 : 5-Dimethyl*cyclo*hexane-1 : 3-dione (DMCH) as a reagent for univalent mercury.** C. Duval and C. Wadier (*Compt. Rend.*, 1955, **240**, 433–435).—5 : 5 - Dimethyl*cyclo*hexane - 1 : 3 - dione (DMCH) is a specific reagent for the detection of Hg at pH 1·5 to 2, producing a greyish or brownish ppt. sol. in HNO_3. A qual. micro-test can detect a min. of 8·7 μg of Hg per 0·05 ml; in the presence of NaF or of 1 per cent. of $AgNO_3$ the sensitivity is increased to 4 μg and 2 μg per drop, respectively. As the composition of the ppt. is variable (owing to the presence of free Hg), a quant. determination of Hg is possible only by redissolution of the ppt. in HNO_3 and use of one of the usual methods.

W. J. BAKER

1776. **A colour test for borates.** C. M. P. Wirth (*Chemist Analyst*, 1954, **43** [4], 101).—Borates in solution can be identified by the colour produced when polyvinyl borate reacts with iodine in aq solution. *Procedure*—Add 5 drops of H_2SO_4 or HCl to the sample, mix well and add 1 ml of dil. aq. polyvinyl alcohol. Mix well and add 1 to 2 drops of 0·1 *N* iodine. In concentrations $>$ 6 mg per 5 ml, a blue colour develops. In lower concentrations, a red or red - violet colour is given. If the 0·1 *N* iodine is added before the acid and alcohol, a green colour develops. Chlorobutanol, dextrin, glucose and zinc sulphate do not interfere.

G. B. THACKRAY

1777. **Micro-titrations with ethylenediaminetetra-acetic acid. XIII. A new method of determining aluminium.** H. Flaschka and H. Abdine (*Mikrochim. Acta*, 1955, [1], 37–43).—The theory and difficulties of determining Al by compleximetric methods are discussed and a method is described. Add an excess of Complexone III soln. (3·723 g per litre) to the weakly acid Al soln. Neutralise to methyl red and add 10 per cent. v/v of buffer (pH 5). Boil for 1 to 2 min. and cool. Add a drop each of 3:3′-dimethylnaphthidine in 1 per cent. acetic acid and aq. ferricyanide soln. (0·1 per cent.). Titrate with zinc chloride (0·01 *M*) to a violet colour. Bring to a yellow colour with a few drops of Complexone III soln. then titrate to violet again. Repeat 2 to 3 times and take the average of the results. *Calculation*: $[V_k.N_k - (V_s - I)N_s] \times 26{\cdot}97 \equiv$ mg of Al, where V_s and V_k are the vol. (ml) of zinc and Complexone soln., respectively, and N_s and N_k are their molar concn.; I is the indicator correction. The method is accurate to ± 0·03 ml of 0·01 *M* standard soln. Ca and Mg do not interfere at pH 5. The method also gives Fe + Al. If both are present the Fe can be determined separately on an aliquot, and the Al calculated by difference.
K. J. GARDNER

1778. **Micro-titrations with ethylenediaminetetra-acetic acid. XIV. The determination of some rare earths.** H. Flaschka (*Mikrochim. Acta*, 1955, [1], 55–60).—The complexiметric determination of Y, Sm, Pr and Gd is described. Three methods are given: (*a*) direct titration at pH 8 to 9 in the presence of tartaric or citric acid at boiling point, with Eriochrome black T as indicator; (*b*) back-titration of an excess of EDTA at pH 8 to 9, in the cold, with standard Zn soln., in the presence of Eriochrome black T; (*c*) back-titration of an excess of EDTA with standard zinc soln. at pH 5, with ferri-ferrocyanide - 3:3′-dimethylnaphthidine as redox indicator. The last method can be used in the presence of alkaline earths. The accuracy is within 0·01 to 0·02 ml of 0·01 *M* standard soln.
K. J. GARDNER

1779. **Improved sensitivity of the iodine pentoxide method for the estimation of carbon monoxide.** H. G. Glover (*Mikrochim. Acta*, 1955, [1], 5–10).—Small amounts of CO in air can be estimated by using the reaction $I_2O_5 + 5CO = I_2 + 5CO_2$, the iodine being determined by absorption in aqueous KI soln. and titrating. A method of absorption in silica gel is described, in which the sensitivity is increased from 3 to 0·1 μg of CO. Silica gel is cleaned by boiling in conc. HNO_3 followed by washing in water and drying. The 60 to 120 B.S. sieve fraction is re-dried at 250° C. The gas containing the iodine vapour is passed through 50 mg of this gel in a special holder; this is sufficient for up to 300 ml of gas at 50 ml per min. About 0·07 ml of 1 per cent. starch solution containing a little KI is added and the iodine is titrated against *N*/560 sodium thiosulphate. The apparatus is described in detail. The technique is particularly suitable for up to 10 p.p.m. of CO and for checking apparatus blanks.
K. J. GARDNER

1780. **Inaccuracies in the determination of carbon monoxide in gas by the iodine pentoxide suspension method.** H. Reens (*Gas, s' Grav.*, 1954, **74**, 96–97).—Tests indicate that the suspension of I_2O_5 in 6 per cent. oleum often used for CO determination gives very inaccurate results if hydrocarbons are present. In the presence of CO there is some attack on hydrogen by the suspension. Deviations amount to as much as 40 per cent. of the actual CO value. It is recommended that the method should be discarded.
CHEM. ABSTR.

1781. **Determination of carbon monoxide with cuprous sulphate - 2-naphthol suspensions.** H. Reens (*Gas, s' Grav.*, 1954, **74**, 98–99).—Previous difficulties with Cu_2Cl_2 for determining CO are overcome in the following method. Prepare Cu_2O from 100 g of cupric acetate in 1 litre of boiling water by adding 60 g of glucose in 400 ml of hot water and boiling for 5 min. Decant, wash the residue with water and with ethanol and dry for 25 min. at 90° to 100° C. Mix 20 g of this oxide with 25 g of 2-naphthol and 25 ml of water, triturate in a mortar and add gradually 200 ml of conc. H_2SO_4. Filter through glass-wool. The gas in an Orsat apparatus is brought into contact with this solution four times for one minute. The capacity for CO of the solution is 10 to 18 ml of CO per ml. Oxygen and unsaturated hydrocarbons are also absorbed, but not saturated hydrocarbons.
CHEM. ABSTR.

1782. **Microchemical detection of cyanides by means of thionaphthenequinone oximes and isatin-β-oxime.** V. Hovorka and J. Morávek (*Bull. Int. Acad. Tchéque Sci. Cl. Sci. Math., Natur. et Méd.*, 1953, **51**, 301–303; *Referativnýǰ Zh., Khim.*, 1954, Abstr. No. 40,025).—In the presence of Fe^{++}, alkali cyanides give intense blue, green and carmine colours with thionaphthenequinone α-oxime, thionaphthenequinone β-oxime and isatin-β-oxime, respectively; with isatin-α-oxime, a faint grey - green colour is produced. To detect CN', 1 drop of a 5 per cent. soln. of $FeSO_4$, 1 drop of a 5 per cent. Na K tartrate soln., 1 drop of a 1 per cent. ethanolic soln. of the reagent and 1 drop of the test soln. are mixed, in that order, on a tile. The sensitivity limit is 1 in 10,000. Chloride, Br', I', S_2O_3'', CNO', NO_3', SO_4'', tartrate, citrate, F', Ba^{++}, Sr^{++}, Ca^{++} and Mg^{++} do not interfere. Ammonium ions and CNS' reduce the sensitivity. Ferrocyanide, ferricyanide, Ag^+, Hg^+, Hg^{++}, Tl, Cu, Cd, Bi, Sn^{++}, Sb^{+++}, Co, Ni, Zn, Fe^{+++}, Al and UO_2^{++} interfere. If interfering cations are present, the sample is treated with H_2SO_4 in a crucible covered with a watch-glass, or in Feigl's apparatus, and the HCN produced is absorbed in Na_2CO_3 soln.
E. HAYES

1783. **Some possible errors in the determination of alkalis in silicates.** W. Geilmann and A. Gänssle (*Glastechn. Ber.*, 1954, **27** [3], 80–88).—In the usual methods of extraction for the determination of alkalis in silicates, losses of alkali can occur. Losses are also found in the further treatment of the extraction solutions, owing to adsorption on the ppt. The losses have been studied by incorporating ^{86}Rb (1 per cent.) and ^{134}Cs (6 per cent.) in soda glass by melting. The methods of Koenig (*Ind. Eng. Chem., Anal. Ed.*, 1935, **7** [5], 314) and of Lawrence Smith (A. W. Groves, "Silicate Analysis," Allen and Unwin, London, 1951) were compared. Losses in extraction were substantially reduced by using a covered vessel. Losses also occurred in the separation of sulphate, in adsorption on other ppt. and in the removal of ammonium salts. Suggestions are made for reducing these losses, which are similar by the two methods, but in Smith's method the chief losses occur in the extraction.
N. E.

1784. **Determination of free-silica content [of silicate materials] by decomposition with phosphoric acid.** K. G. Schmidt (*Ber. dtsch. keram. Ges.*, 1954, **31** [12], 402–403).—The silicates are dissolved in a

mixture of $H_4P_2O_7$ and HPO_3 prepared by heating (in vitreous silica ware) conc. H_3PO_4 (84·5 per cent.) at 250° C until bubbling ceases. *Procedure*—A vitreous silica crucible containing 0·1 g of sample and 8 to 10 ml of prepared acid is placed in a block of copper heated to 275° C; the mixture is stirred continuously at 250° C for 10 to 20 min. The crucible is cooled in water, the contents are diluted with 50 to 60 ml of hot water and filtered hot. The addition of 20 mg of ammonium tartrate and one crystal of tartaric acid before filtration may sometimes be necessary to prevent the separation of gelatinous $Si(OH)_4$. If persistent $Si(OH)_4$ prevents satisfactory filtration, a fresh sample is decomposed with acid, as above, but 10 ml of HBF_4 (27° Bé) should be included in the diluting water and the mixture set aside for 1 hr. before filtering. The residue on the filter is washed once with hot acidified (acetic) water and three times with hot 4 per cent. HCl (for removal of $Al^{\cdot\cdot\cdot}$ and $PO_4^{\prime\prime\prime}$) and finally with cold acidified (acetic) water. The paper and residue are ignited at 850° C and the SiO_2 is volatilised with HF - H_2SO_4. If the final residue in the crucible exceeds 0·75 mg, the whole procedure is repeated with a longer reaction time. If there is now an undue residue, a correction must be made for the SiO_2 arising from any undissolved silica-containing compounds (*e.g.*, mullite, parts of some felspars, SiC, etc.), which are identified under the microscope before the treatment with HF. Difficulties due to colloidal precipitates usually arise with materials rich in glassy phase, bentonite, alkaline earths or sesquioxides. The results for a range of clays agreed well with the free-silica content determined by a combination of ultimate and rational chemical analysis and X-ray and differential thermal analysis. J. A. SUGDEN

1785. **The quantitative extraction of germanium tetrabromide with ether. I.** I.-M. Ladenbauer, O. Slama and F. Hecht (*Mikrochim. Acta*, 1955, [1], 118–122).—A new method for the quant. estimation of Ge is described. Place not more than 25 ml of neutral soln. into the continuous extractor described previously (Hahofer and Hecht, *Anal. Abstr.*, 1954, **1**, 2404). Treat with 100 ml of a 48 per cent. HBr soln. which is saturated with ether and KBr. Extract for 5 hr. with 80 ml of ether. Then shake the ethereal soln. several times with a few ml of HBr soln. followed by 10 to 80 ml of H_2O in small portions, the total amount being dependent on the Ge originally present. Determine the Ge on an aliquot gravimetrically or colorimetrically by the method of Cluley (*Analyst*, 1951, **76**, 523). When applying this method to zinc blende, saturated titanium tribromide was added before extraction. This acts as reducing agent and prevents extraction of Fe. K. J. GARDNER

1786. **Semi-quantitative paper-chromatographic determination of germanium with phenylfluorone.** I.-M. Ladenbauer (*Mikrochim. Acta*, 1955, [1], 139–145).—In an extension of an earlier method (Ladenbauer *et al.*, *Anal. Abstr.*, 1954, **1**, 2377, 2378) the Ge soln. is applied to Schleicher and Schüll 2043b (rough) paper and developed in butanol saturated with 10 per cent. HBr. After drying, the Ge is made visible by spraying with a 0·05 per cent. soln. of 2:3:7-trihydroxy-9-phenyl-6-fluorone (phenylfluorone) in ethanol - H_2SO_4 (d = 1·19) (75 + 25). The outline of the spot is marked in pencil immediately after spraying, and Ge is determined by measuring the area. A calibration curve derived from 3 known amounts must be made with every estimation. For easy separations 10 μl of solution are run for 22 hr.; for difficult ones (*e.g.*, if Pb is present) 5 μl are run for 40 hr. Ge was separated from the 15 most important elements of groups II and III and estimated with a mean accuracy of ± 6 per cent. K. J. GARDNER

1787. **Voltammetric estimation of lead with potassium ferrocyanide.** B. Khosla, H. C. Gaur and N. A. Ramaiah (*Curr. Sci.*, 1954, **23** [11], 361).—The manual polarographic arrangement with dropping-mercury electrode was used to extend the method of an earlier publication (*J. Sci. Ind. Res., B., India*, 1954, **13**, 304) to the quantitative determination of small amounts of lead. $Pb^{\cdot\cdot}$ can be determined within an error of ± 1 per cent. and the method can be employed in the presence of $Ba^{\cdot\cdot}$ or $Ca^{\cdot\cdot}$. R. J. MAGEE

1788. **Estimation of thorium by organic reagents. III. Use of 2:4-D in the separation of thorium from zirconium, titanium and iron and their co-determinations.** S. K. Datta and G. Banerjee (*J. Indian Chem. Soc.*, 1954, **31** [10], 773–778).—Methods are reported for the use of 2:4-D [2:4-dichlorophenoxyacetic acid] (**I**) in the separation of Th from Fe^{II}, Fe^{III}, Zr and Ti and for the co-determination of these metals. The separation and co-determination of Th and Ti involve (*i*) neutralisation of the solution to Congo red with dil. NH_3 soln., (*ii*) addition of 5 to 22 ml of 3·5 *N* HNO_3 and 0·5 g of ammonium acetate, (*iii*) dilution to 50 ml and pptn. of the zirconium with **I** (1·5 per cent. soln.), (*iv*) washing the zirconium ppt. with a 0·2 per cent. solution of **I** and then with 0·1 *N* HNO_3 and ignition to the oxide, (*v*) evaporation of the filtrate and washings to 40 ml, and making neutral to Congo red by adding dil. NH_3 soln., and (*vi*) addition of an excess of **I** solution. The resulting thorium ppt. is filtered off, washed and ignited to ThO_2. This method may be used to separate Th from Fe^{II} and Ti, but Fe^{II} requires a double pptn. The application of this method to the separation of Th from Fe^{III} requires the additional use of ammonium carbonate and involves (*i*) neutralisation of the solution to Congo red, (*ii*) addition of 1 g of ammonium acetate and dilution to 50 ml, (*iii*) addition of an excess of a warm 1·5 per cent. solution of **I**, (*iv*) filtration of the resultant mixed ppt. and washing with 50 per cent. ethanol, (*v*) addition of a 10 per cent. solution of ammonium carbonate with constant stirring for 10 min., avoiding excess, (*vi*) filtration of the solution from the ferric hydroxide after being set aside overnight, (*vii*) washing of the ppt. by decantation with 2 per cent. ammonium carbonate soln., washing on the filter with tepid water and collection of the filtrate and washings, (*viii*) dissolution of the ferric hydroxide in a min. amount of HCl and re-precipitation with 10 per cent. ammonium carbonate soln., (*ix*) repetition of filtration and washing, (*x*) gravimetric determination of Fe, (*xi*) boiling the combined filtrate for ≃ 1 hr., decomposing the Th salt to the hydroxide by carefully neutralising to Congo red, (*xii*) separate filtration and washing of the precipitates and ignition to the oxide. A different process is required for separation of Th from a mixture of Zr, Fe^{III} and Ti in solution, namely (*i*) removal of Fe by the Gooch process with tartaric acid, (*ii*) evaporation of the filtrate nearly to dryness, (*iii*) destruction of the tartaric acid present by fuming with conc. H_2SO_4 and HNO_3, (*iv*) dissolving the residue in H_2O, (*v*) separation of Zr and Th with **I** as described above, (*vi*) gravimetric determination of Ti in the final filtrate. L. F. TAYLOR

1789. **Estimation of thorium by organic reagents. IV. Volumetric estimation by anthranilic acid and gravimetric estimation by 5-iodoanthranilic acid.** S. K. Datta and G. Banerjee (*J. Indian Chem. Soc.*, 1954, **31** [10], 779–783).—The volumetric determination of Th is effected by quant. bromination, with *N* $KBrO_3$, of Th anthranilate (pptd. by the Murthy-Rao method, *Ibid.*, 1950, **27**, 549) dissolved in aq. HCl (1 + 1). The soln. should contain 0·5 to 1 g of KBr and a few drops of 1 per cent. indigo carmine to indicate complete pptn. of dibromoanthranilic acid. The titration is completed iodimetrically; 1 ml of *N* $KBrO_3 \equiv 0{\cdot}029015$ g of Th. The gravimetric determination of Th is effected by addition of a 1 per cent. aq. soln. of either the sodium or ammonium salt of 5-iodoanthranilic acid (preparation described) to a boiling soln. containing $Th^{\cdots\cdot}$ at a pH > 4, in the presence of 0·5 g of ammonium acetate. The ppt. is washed with 0·2 per cent. aq. soln. of the precipitating reagent, then with hot water, and ignited to ThO_2. This method can be used to separate Th from cerite earths in monazite extracts, two pptn. being made. All other metals, except the alkaline earths, should be absent. The composition of the Th compound of 5-iodoanthranilic acid is indefinite.

W. J. Baker

1790. **Determination of nitrogen by the nuclear reaction $^{14}N(d,n)^{15}O$.** P. Süe (*Compt. Rend.*, 1955, **240** [1], 88–90).—A micro-determination of nitrogen (6 to 30 μg) with an accuracy of ± 20 per cent. depends on the observed relation between mass of N, activity of ^{15}O, and deuteron intensity. Results for fused KNO_3 are reported. The salt, supported on aluminium foil (0·05 mm), is irradiated for 10 min. in a deuteron beam of ≃ 2 to 3 μA; after irradiation, the sample is quickly powdered and measurements by counter are made of the activity of ^{15}O (half-life 2·1 min.) and, about 45 min. later, of ^{42}K (half-life 12·4 hr.). One thousand pulses due to ^{15}O are equivalent to 0·88 μg of nitrogen, whilst 1 mg of potassium corresponds to one thousand pulses due to ^{42}K. Sources of error are: the activity of ^{17}F (half-life 1·1 min.), the difficulty of standardising the conditions of irradiation, and interference by (*d*, *p*) reactions of Al, Ag, V, Tl or Nb present as impurities. The procedure can detect 1 μg of nitrogen, or 1 p.p.m. in a metal. W. J. Baker

1791. **Spectrophotometric study of the ammonia-pyridine-pyrazolone method [for the determination of nitrogen in steel or soil].** J. B. Lear (*Dissert. Abstr.*, 1954, **14** [10], 1520).—The application of this method to the determination of N in steel and soil is studied, together with the mechanism of the reaction. The N present is converted into ammonia, which is then determined with the pyridine-pyrazolone reagent. The original paper gives results of 23 analyses of steel for both soluble and insoluble N, and also proposed procedures for the determination of total N in soils and NH_3 in soil extracts. The lack of reliably analysed soil samples has prevented a study of the accuracy of this method as applied to soils, but results indicate that the method should give satisfactory data.

S.C.I. Abstr.

1792. **Detection and photometric determination of nitrite with thioglycollic acid.** M. Ziegler and O. Glemser (*Z. anal. Chem.*, 1955, **144** [3], 187–191).—Amounts > 30 μg of NO_2' are detected by thioglycollic acid, and determined photometrically in u.v. light at 355 mμ, after separation from aq. solution by various solvents. The best solvent is a mixture of di*iso*propyl ether-pentanol-acetone (10 : 10 : 1). An accuracy of ± 3 per cent. is attainable. D. R. Glasson

1793. **Separation and quantitative determination of condensed phosphates by means of paper chromatography.** K. W. Gerritsma and J. C. Frederiks (*Chem. Weekbl.*, 1955, **51** [11], 197–201).—Mixtures of orthophosphates and condensed phosphates can be separated by partition chromatography. Alkaline or acidic developers can be used, the former comprising a mixture of 30 ml of *iso*propanol, 30 ml of ethanol, 39 ml of water and 1 ml of 25 per cent. ammonia soln., and the latter 75 ml of *iso*propanol, 25 ml of water, 5 g of trichloroacetic acid and 0·3 ml of 25 per cent. aq. ammonia. Ortho-, pyro-, tripoly-, trimeta- and tetrameta-phosphates and high-polymer phosphates can be satisfactorily separated. After elution of the separated phosphates from the paper with dilute aq. ammonia, the amount is determined colorimetrically.

A. J. Mee

1794. **A rapid method for the determination of arsenic in iron, steel and iron ores by means of a photo-electric filter photometer.** S. Hirano and D. Ishii (*Japan Analyst*, 1953, **2** [1], 28–31).—The turbidimetric method for determining As (Tsyvina *et al.*, *Zavod. Lab.*, 1939, **14**, 28) has been applied to the rapid analysis of iron. The reduction of As to the elementary state with hypophosphorous acid can be completed in $HClO_4$-HCl soln. within 20 min. in a boiling-water bath. This reduction is catalysed by iodide (J. Langlois *et al.*, *Bull. Sci. Pharmacol.*, 1938, **45**, 482) so that it takes 7 min. at room temp. The colloidal soln. of As obeys Beer's law within the range 0·5 to 15 μg per ml. *Procedure*—The sample (0·2 g) is treated with dil. HNO_3 (1 + 1) (3 ml) in a small beaker and heated with 60 per cent. $HClO_4$ (5 ml) until white fumes of $HClO_4$ are obtained. It is then dissolved in H_2O (5 ml) and $Fe^{\cdots}$ is reduced to $Fe^{\cdot\cdot}$ with $SnCl_2$. Hypophosphorous acid (15 ml) is added and the solution made up to 30 ml. The soln. is kept in a boiling-water bath (20 min.) and the light absorption is measured, after cooling. Alternatively, 20 per cent. NaI soln. (3 ml) can be added to the soln. before making up to 30 ml. Hypophosphorous acid is prepared by dissolving $NaH_2PO_2.H_2O$ (50 g) in H_2O (50 ml), adding conc. HCl (500 ml) and filtering off the NaCl. The presence of Se, Mo and coloured ions, such as Co and Ni, affects the result, but Mn, Cu and Fe do not. The whole analysis can be completed within 25 min. K. Saito

1795. **Separation of tantalum and niobium by liquid-liquid extraction.** J. R. Werning and K. B. Higbie (*Ind. Eng. Chem.*, 1954, **46** [12], 2491–2494).—Tantalum can be preferentially extracted (≃ 60 per cent. yield) by conc. HCl (approx. 12 *N*) from a soln. of anhydrous tantalum and niobium pentachlorides in *iso*butyl methyl ketone-di*iso*butyl ketone (2 + 5, by vol.). In the presence of large amounts of $FeCl_3$, *e.g.*, a 1 : 1 : 2 by wt. mixture of the three chlorides, the wt. of $TaCl_5$ extracted is increased greatly (yield ≃ 95 per cent.), whilst the amount of Nb extracted is increased only slightly. The Nb and Fe remaining in the organic phase are separated by digesting the mixture for ≃ 2 min. with hot dil. H_2SO_4, decanting the aq. phase, digesting it for ≃ 1 hr., filtering off and washing the ppt. with H_2O and acetone to yield $NbCl_5$ containing ≃ 0·1 per cent. of Fe. The filtrate is neutralised

with aq. NH_3 (28 per cent.) to ppt. $Fe(OH)_3$ containing $\simeq$ 1 to 2 per cent. of Nb. The original ketones can also be recovered simply. Procedures for effecting single- or multi-stage (3 or 4) extractions from solutions of the crude chlorides (*e.g.*, from tin slags), or iron-free chlorides, are described fully. The process is especially applicable to low-grade tantalum and niobium ores containing a high percentage of Fe. W. J. BAKER

1796. **Polarographic determination of oxygen and hydrogen peroxide.** J. C. Fittipaldi (*Rev. Fac. Ing. Quím., Argentina*, 1952–53, **21–22** [35], 123–131).—A simple apparatus, made from readily available materials, is described for use in the polarographic method for the determination of dissolved oxygen or hydrogen peroxide. H. PRITCHARD

1797. **A volumetric determination of oxygen in metals.** H. Kamata, H. Yamamoto and Y. Iida (*Japan Analyst*, 1953, **2** [1], 36–37).—When a metal sample is heated *in vacuo* ($\simeq$1100° C, 30 min.) in a graphite boat, all the oxygen contained in it is converted into CO and CO_2. The apparatus for the determination of oxygen in metals by the hydrogen reduction method can be used without alteration. The CO and CO_2 are converted into H_2O and CH_4 in a current of hydrogen and with the use of the usual Ni-thoria catalyst. Instead of being absorbed by P_2O_5, the gas is passed through cinnamoyl chloride at 60° to 70° C, producing an equiv. amount of HCl, which is absorbed in water and titrated with standard NaOH soln. The result was in fairly good agreement with those obtained by the usual hydrogen reduction method. The small fluctuation of the data can be reduced by the improvement of the bubbler for HCl absorption. K. SAITO

1798. **Magnetic measurement of oxygen with the hot-wire system.** H. Krupp (*Chem.-Ing.-Tech.*, 1955, **27** [2], 79–83).—Thermomagnetic methods for the determination of oxygen, which depend on paramagnetic properties, are described. The most important of these methods, which is well suited to continuous determinations, is described in detail. In this method, the gas passes between the pole pieces of a permanent magnet, where it is blown out by the field as a "magnetic wind" of force that increases with increasing oxygen content. The magnetic wind blows on to an electrically heated wire thereby cooling the wire, and the resulting change in resistance of the wire (as measured by a Wheatstone bridge) is used to determine the oxygen content of the gas. The theory of the system and the desiderata for an instrument of optimum efficiency are discussed. The "Magnos 5" oxygen recorder, which embodies these desiderata, is described in detail. H. L. WHITEHEAD

1799. **Rapid determination of sulphur in sheet glass [by a combustion method].** M. Takahashi (*Japan Analyst*, 1953, **2** [1], 26–28).—To avoid the complicated gravimetric method, a combustion method followed by volumetric determination of the SO_2 (after oxidation with H_2O_2) with NaOH is proposed. The method is similar to that for sulphur determination in iron and steel. For the flux, both B_2O_3 and Pb_3O_4 can be used, provided that their amount is more than twice that of the sample (0·3 g). The temp. of the combustion tubes should be kept at 1350° to 1400°C and oxygen passed through it for 10 min. (350 to 400 ml per min.). A porcelain boat (of high Al_2O_3 content) is a suitable container. The concn. of H_2O_2 is 0·1 to 0·5 per cent. The result agrees well with that obtained by gravimetric analysis; the whole procedure takes $\simeq$ 15 min. K. SAITO

1800. **Detection of sodium dithionite and sodium formaldehydesulphoxylate by means of spot tests.** F. Feigl and L. Hainberger (*Mikrochim. Acta*, 1955, [1], 105–109).—Add a few mg of the substance under test to a drop of saturated ethanolic *o*- or (preferably) *p*-dinitrobenzene in conc. aq. NH_3. Sodium dithionite produces an immediate blue-violet or orange coloration (for the ortho or para compound, respectively) at room temperature; sodium formaldehydesulphoxylate (**I**) gives only a very weak coloration after several minutes. No colour is produced with **I** at ice temperature, but an immediate colo[illegible] on heating. The sensitivity is 3 μg. The presence of **I** can be confirmed by testing for formaldehyde. K. J. GARDNER

1801. **Rapid photometric determination of tellurium in copper-tellurium alloys.** W. Nielsch and G. Böltz (*Z. Metallkunde*, 1954, **45**, 380–386).—A rapid photometric tellurium assay for use in a smelter is described. The reagents are 7·5 *N* HNO_3 (*A*) and thiourea soln. (*B*) (100 g dissolved by heating, below 70°C, in 500 ml of water and made up to 1 litre). *Procedure*—Dissolve 1 g of filings in a 300-ml Erlenmeyer flask with 25 ml of *A*, transfer to a 100-ml calibrated flask and dilute to volume. To 10 ml of this soln. in another 100-ml flask add 20 ml of *B*, make up to vol. and determine with a 380-mμ filter in a photo-electric colorimeter, using the Hg 366-mμ line as source. Beer's law is shown to hold for 1·5 to 24 μg of Te per ml. Traces of Fe are complexed by H_3PO_4. CHEM. ABSTR.

1802. **Photometric determination of tellurium by thiourea.** W. Nielsch (*Z. anal. Chem.*, 1955, **144** [3], 191–192).—An extension of an earlier method (*Anal. Abstr.*, 1955, **2**, 1801) permits the determination of Te in smaller amounts (0·01 to 0·0001 per cent.) in alloys containing Cu and Se. D. R. GLASSON

1803. **Reactions of dithiocarbamates with molybdates.** L. M. Kul'berg and A. G. Kovaleva (*Dokl. Akad. Nauk, SSSR*, 1954, **98**, 79–81).—Red ppt. are given by molybdates (0·003 to 100 μg of Mo) with dithiocarbamates $NRR'\cdot CS_2H$ (R = H; R' = H, NH_2, $CH_2\cdot CH_2\cdot NH\cdot CS_2H$, *iso*-$C_4H_9$, $CH(C_6H_5)_2$, C_6H_5, *m*- and *p*-$C_6H_4CH_3$, *p*-$C_6H_4\cdot C_6H_5\cdot NH\cdot CS_2H$ or CH_2CH_2OH; R = R' = CH_3, *n*-C_4H_9, *iso*-C_4H_9, *iso*-C_5H_{11}, *n*-C_5H_{11} or CH_2CH_2OH). The ppt. have the composition $MoO_3[NRR'\cdot CS_2H]_2$. Similar reactions are given by xanthates and dithiophosphates, which suggests that the functional analytical group is $A\cdot MS_2H$, where M = C or P, A = NRR' or OR, and R = H, alkyl or aryl. R. TRUSCOE

1804. **Quantitative determination of fluoride ions by a potentiometric method.** Sh. T. Talipov and I. L. Teodorovich (*Dokl. Akad. Nauk Uzb. SSR*, 1953, [8], 32–34; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 40,030).—The precision of the method for the potentiometric determination of NaF by titration against an aluminium salt with the quinhydrone electrode (Tananaev, *Zavod. Lab.*, 1933, [10], 13) is studied. The mean square error is $\pm$ 1·9 per cent. E. HAYES

1805. **Use of ion-exchange resins for determination of atmospheric fluorides.** J. P. Nielsen and A. D. Dangerfield (*Arch. Ind. Hlth.*, 1955, **11** [1], 61–65).—The method described involves the absorption of the fluoride in a water bubbler (containing

a little $BaCl_2$, if S compounds are present) and then passing the solution over an anion-exchange resin (*e.g.*, Duolite A 41), followed by elution with a small volume of sodium hydroxide solution to isolate the fluoride from interfering cations. Distillation for the isolation of fluorides, and variable reagent and system blanks are eliminated. The F is titrated in the usual way with thorium nitrate soln. Data on pure solutions of HF and fluoride salts show that these substances can be added to, and recovered quantitatively from, the ion-exchange column. The accuracy below 20 μg is ± 1 μg. I. JONES

1806. **The determination of fluorine in Piltdown and related fossils.** C. R. Hoskins and C. F. Fryd (*J. Appl. Chem.*, 1955, **5** [2], 85–87).—Earlier determinations of fluorine in fossils are reviewed and found to be unsuitable for the small amounts (2 to 5 mg) of bones and teeth of the Piltdown specimens available. A suitable technique, which involves a final titration with thorium nitrate in a Nessler tube, is described in detail. *Procedure*—An alkaline sample in 20 ml of water and 2 drops of silver perchlorate solution is distilled into a receiver containing 2 drops of phenolphthalein, 2 drops of 0·1 *N* NaOH and 20 ml of perchloric acid. The temp. (136° to 140° C) is regulated by a Bunsen burner and dropwise addition of H_2O at the rate of distillation, and the distillate is kept alkaline by addition of 0·1 *N* NaOH, when required. The operation is stopped when 75 ml of distillate have been collected. About 5 ml of concentrated alkaline distillate, 1 ml of buffer (50 ml of 4 *M* chloroacetic acid, neutralised with NaOH, added to 50 ml of 4 *M* acid and made up to 200 ml) and 6 drops of alizarin indicator made up to 50 ml in a Nessler tube are titrated with an aq. thorium nitrate solution (adjusted so that 0·2 ml produces the required colour) to match a control solution in an identical Nessler tube. Titres of 0·25 to 1·00 ml of 0·002 *M* are recommended. The results vary from < 0·01 per cent. of F in dentine, Piltdown II, to 0·21 ± 0·04 per cent. of F in a portion of skull.

S.C.I. ABSTR.

1807. **Micro-determination of iodine by the catalytic method.** F. Lachiver (*Ann. Chim.*, 1955, **10** [1–2], 92–134).—As a preliminary to studying the effect of iodine on the reduction of arsenous acid (**I**) by ceric sulphate (**II**), the optical density of solutions of **II** at 440 mμ was determined; Beer's law is not obeyed, but the log (optical density) is a linear function of log (concn. of **II**) over the range 0·001 *N* to 0·01 *N*. Slight variations in slope are produced by variations in temp. and acidity. The rate of reaction of **I** with **II**, studied by measuring the optical density at constant intervals of time, is first-order in relation to **II** for concn. of **I** from 1 to 3 times that of **II**. The velocity constant is a linear function of the concn. of iodine, and is affected by the presence of Cl′ or Br′, which in addition have small independent catalytic effects. It is suggested that they remove substances which inhibit the iodine-catalysed reaction. The variations of the catalytic coefficients with acidity, temp., and concn. of Cl′ and Br′ are determined. One effect of the presence of Cl′ or Br′ is to increase the range of concn. of **I** and **II** in which the reaction shows first-order behaviour. Optimum conditions are determined for the estimation of iodine, by measuring the velocity constants in the presence and absence of the iodine-containing substance; a reaction vol. of 4 to 5 ml. in a cuvette is suitable. The temp. should be held at 35° ± 0·1° C for small quantities of iodine, but may be 25° C for larger amounts. The solution should be 0·009 *N* in $Ce(SO_4)_2$, 0·5 *N* in H_2SO_4, and contain 0·5 to 1 mg per ml of Cl′ or Br′. The best iodine concn. is 5 to 15 μmg per ml, but the reaction can be followed at iodine contents from 0·1 to 35 μmg per ml. Iodate has no catalytic effect. The reaction is inhibited by Hg, Pb, Ag, CN′ and CNS′, and strongly catalysed by Os. Because of these factors, and since in unknown or biological materials iodine may be present as a complex, or other unsuitable form, it is essential to mineralise the material before using the catalytic method. H. P. PAGET

1808. **Ultra-violet spectrophotometric determination of iron with ethylenediaminetetra-acetic acid.** Y. Uzumasa and M. Nishimura (*Bull. Chem. Soc. Japan*, 1955, **28** [1], 88–89).—In acid medium, Fe··· forms a complex with ethylenediaminetetra-acetic acid (**I**) which gives intense absorption in the u.v. region. The test soln. was prepared so that it was 0·1 *N* in HCl, 0·001 *M* in **I**, and the Fe··· concn. was between 5×10^{-5} and $1{\cdot}5 \times 10^{-4}$ *M*. Absorption was measured in 1-cm silica cells at 260 mμ. H_2SO_4 may be used as well as HCl and the absorption remains const. over an acid range of 0·001 to 0·3 *N*.

H. F. W. KIRKPATRICK

1809. **Colorimetric determination of iron with salicylaldehyde - glycinehydroxamic acid.** A. Mukherjee (*Naturwissenschaften*, 1955, **42** [5], 127).—The Schiff's base derived from salicylaldehyde and glycinehydroxamic acid gives an intense violet coloration with Fe··· and can be used to estimate traces. The maximum absorption for the colour of the complex formed lies between 535 and 550 mμ at pH 2·35 to 3·34 and between 460 and 510 mμ at pH 5·06 to 10·26. S. R. NEUBERGER

1810. **New photometric determination of iron in the analysis of copper [-base] alloys. I. Principles and procedure.** W. Nielsch and G. Böltz (*Metall*, 1954, **8**, 374–378).—The metal is determined as the yellow ferric tartrate, using $NaIO_4$ as stabiliser to suppress reduction to a colourless solution in the presence of u.v. light. A Zeiss Elko II photometer is employed, with monochromatic mercury light (366 mμ), S38E filter, u.v. condenser, and a 2-cm cell. Preliminary experiments are described. Pure Fe (60 mg) was dissolved in 2 ml of HNO_3 (1 + 1), the solution was diluted to 1 litre, 10 ml of which were transferred to a 100-ml calibrated flask, and 2 ml of 1 per cent. aq. $NaIO_4$ and 20 ml of 25 per cent. tartaric acid were added, together with various amounts of HNO_3 or NaOH to give a pH between 0·99 and 12·62. The vol. was made up to 100 ml with distilled water, and the extraction values were determined. Optimum results were obtained at pH 1·52 to 3·05 and 3·50 to 7·10. In actual determinations separate calibration curves are necessary, as the corresponding transmission values were found to be 45·80 and 43·00 per cent., respectively. The influence of $NaIO_4$ content, temp. and time of colour development (15° to 35° C, and 10 min. are suitable), tartaric acid content, various buffers (NH_4NO_3, $K_2B_4O_7$, NaCl), and of the presence of various amounts of other metals and of $Na_4P_2O_7$ are discussed. METAL ABSTR.

1811. **Determination of iron and titanium in admixture. (Application of masking action to volumetric analysis.)** S. Suzuki, K. Harimaya and M. Ueno (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [12], 1287–1290).—Ferrous iron can be determined iodimetrically (back-titration with $Na_2S_2O_3$) in the presence of NH_4HF_2 or tartrate only

in neutral soln., but $Ti^{\cdots}$ can be titrated iodimetrically in acidic soln. even in the presence of $Fe^{\cdot\cdot}$ and a small amount of NH_4HF_2. This fact can be utilised for the quant. analysis of a mixture of these ions or of $Fe^{\cdots}$ and $Ti^{\cdots\cdot}$, after reduction with zinc amalgam. Experimental conditions are examined and some examples are shown. K. SAITO

1812. **Complexiметric titrations (chelatometry). XI. 1 : 2-Diaminocyclohexane-NNN′N′-tetra-acetic acid as volumetric reagent. Stepwise determination of iron and manganese (magnesium, calcium); determination of copper in presence of iron, nickel, cobalt and manganese.** R. Přibil (*Coll. Czech. Chem. Comm.*, 1955, **20** [1], 162–169).—The properties of the complexes of a number of metals with disodium 1:2-diaminocyclohexane-NNN′N′-tetra-acetate (**I**) are discussed with relation to their volumetric determination. For the stepwise determination of Fe and Mn, titrate the Fe with **I** at pH 2 with salicylic acid as indicator, then titrate the Mn with Eriochrome black T as indicator after adding hydroxylamine and triethanolamine and making the soln. alkaline; the stepwise determination of Fe and Mg or Ca is similar, but no triethanolamine is needed. For the determination of Cu in the presence of Fe, Ni, Co or Mn, add an excess of **I** and back-titrate with $MgSO_4$ to give total metals; then add KCN and H_2O_2 (or hydroxylamine in the case of Fe) to break down the Cu complex, and titrate the **I** released. [This is a translation into German of a paper originally published in *Chem. Listy*, 1955, **49**, 179.] A. R. ROGERS

1813. **Spectrographic determination of small amounts of cobalt in nickel salts.** F. Čůta and K. Rauscher (*Coll. Czech. Chem. Comm.*, 1955, **20** [1], 180–187).—Small amounts (0·005 to 0·1 per cent.) of Co in Ni salts may be determined with an error of ± 6 per cent. by the method of Rivas (*Brit. Abstr. A*, 1936, 1084) using the 2378·6 A (Co) and 2379·7 A (Ni) lines of the emission spectrum between carbon electrodes. [This is a translation into German of a paper originally published in *Chem. Listy*, 1954, **48**, 1616.] A. R. ROGERS

1814. **Quick moisture method for coal.** V. R. Gray and P. F. Whelan (*Chem. & Ind.*, 1955, [6], 126–128).—The method depends on the heat effect caused by adding conc. H_2SO_4 to the coal sample. The mixing takes place in an unsilvered Dewar flask, and the max. temp. rise is measured. The relationship between temp. rise and moisture content varies with the type of coal, and a nomograph has been constructed to give a direct correlation between these three factors. The accuracy is within 5 per cent., which is sufficient for plant control. A possible difficulty is the dependence on particle size of the coal. A. M. SPRATT

See also Abstracts 1962, 1975.

3.—ORGANIC ANALYSIS

1815. **The removal of nitrogen oxides in the micro-determination of carbon and hydrogen.** T. Mitsui (*Japan Analyst*, 1953, **2** [1], 3–5).—The use of H_2SO_4 saturated with CrO_3 has been recommended by some workers to remove the nitrogen oxides that are produced by the combustion of organic substances in the micro-analysis of C and H. The influence of this liquid absorber is studied with the use of a new type of bubbler which was designed by the author (*Bull. Res. Inst. Food Sci., Kyoto Univ.*, 1952, **8**, 36). When N is present in the amino form, the result is satisfactory, but when present in the nitro form the result for H [absorbed by $Mg(ClO_4)_2$] is affected, showing that the use of this absorbent is not desirable for micro-analysis. K. SAITO

1816. **A rapid micro-analytical determination of halogen in organic substances.** W. Schöniger (*Mikrochim. Acta*, 1955, [1], 123–129).—Weigh 4 to 8 mg of the substance under test on to an ashless filter-paper, fold the paper and suspend it from a platinum wire in an Erlenmeyer flask containing 10 ml of H_2O, 1 ml of KOH soln. (2 *N*) and 3 drops of H_2O_2 (30 per cent.). Fill the flask with O, ignite the filter-paper and close the stopper. Shake several times during 10 min. to absorb the gases. Wash down and boil for 5 min. to destroy the H_2O_2. Acidify with 3 ml of 2 *N* H_2SO_4, re-boil and cool. For the determination of Cl and Br, neutralise with KOH (2 *N*) to methyl red - methylene blue; add 10 ml of saturated mercury cyanate soln. Titrate the OH′ produced with 0·01 *N* H_2SO_4. For the determination of I, the H_2O_2 is omitted from the test described above. After absorption of the gases, 10 ml of a soln. of K acetate (100 g) in acetic acid (1000 ml), containing 4 ml of Br, are added and the excess of Br is reduced with formic acid. Titrate the I in the usual way. The accuracy attained was ± 0·3 per cent. K. J. GARDNER

1817. **Determination of organically combined iodine.** H. Aasness (*Medd. Norsk Farm. Selsk.*, 1954, **16** [7–8], 61–76; [9], 85–105; [10], 112–121).—A detailed study of the methods of determination of iodine in organic compounds is presented. The method recommended is as follows: The compound (about 10 mg of I) is warmed with 1 g of $KClO_3$ and 0·1 g of NaCl, then 2 ml of H_2SO_4 (50 per cent. v/v) are added to the boiling solution. After heating at a temp. not exceeding 230° C until the mixture is nearly dry and colourless, and white fumes appear, 25 ml of water are added and the mixture is boiled for 2 min. One drop of phenolphthalein soln. is added and 2 *N* NaOH to a red colour, then the mixture is neutralised, 1·5 ml of *N* HCl being added in excess. After adding 5 ml of 5 per cent. KI, the solution is titrated with 0·1 *N* $Na_2S_2O_3$. By this method any residual chlorate is transformed to perchlorate, which does not interfere. Insoluble acidic compounds are first dissolved in alkali before the addition of chlorate. Examples are given of the application to iodoacetic acid, di-iodotyrosine, diodone, pheniodol and thyroxine sodium. G. MIDDLETON

1818. **The decomposition of carbon dioxide in contact with copper oxide and a modified combustion method for the determination of nitrogen.** T. Mitsui (*Japan Analyst*, 1953, **2** [2], 117–121).—The decomposition of CO_2 in the combustion tube used in Dumas' micro-method is closely studied, with attention to the purity of Cu and CuO. When the CuO (at 20° to 700° C), which is usually placed at the end of the tube, is sufficiently pure, a significant amount of O is produced (temp. of the main furnace, 870° C). CuO thus shifts the equilibrium $2CO_2 \rightleftharpoons 2CO + O_2$ to the right by reacting with CO, contrary to the conventional belief that hot CuO promotes the association of O and CO to form CO_2. If all the CuO in the tube is substituted by pure Cu, it will absorb O, so that CO is evolved (at 600° to 800° C). Thus the following modification is suggested. The layer of Cu is transferred to the end of the CuO layer and heated at 500° to 600° C in a separate

furnace. A high temp. of the first furnace, which promotes the decomposition of organic substances in the current of CO_2, does not then produce any undesirable effect. K. SAITO

1819. **The micro-analytical determination of nitrogen by means of decomposition with magnesium. II.** W. Schöniger (*Mikrochim. Acta*, 1955, [1], 44–48).—On heating nitrogen-containing organic compounds with Mg in the presence of CO_2, magnesium nitride is formed, the nitrogen in which can be determined by Kjeldahl distillation. Weigh the substance under test (3 to 5 mg) into a decomposition tube (6 mm × 100 mm). Add magnesium powder to a depth of 3 to 4 mm, mix well, then add more Mg to a depth of 40 mm. Add two lots of "dry ice" and evaporate to drive out atmospheric N. During the second evaporation draw out the end of the tube to a capillary, and when all the CO_2 has been liberated seal the tip. Insert this in a tube through which a constant stream of CO_2 is passed. Cut off the tip and heat the charge to redness, starting from the top. Place the contents in water, add dil. H_2SO_4 and boil. Determine the $(NH_4)_2SO_4$ produced by Kjeldahl distillation. Determinations of S, Cl and Br can be made on the same sample, before distillation, by the method described earlier (*Anal. Abstr.*, 1954, **1**, 951). K. J. GARDNER

1820. **Micro-determination of nitrogen, sulphur, chlorine and bromine in organic substances.** M. Večeřa (*Mikrochim. Acta*, 1955, [1], 88–100).—N is determined by the Dumas - Zimmermann method (*Mikrochemie*, 1943, **31**, 42) with an improved apparatus. The substance is burnt in a series of three furnaces in the presence of CO_2. Complete decomposition of liquids is effected by heating with a burner that is moved along the furnace by an electric motor. This prevents vapours from collecting in a cool area and thus escaping decomposition. Sulphur is determined by burning the substance in a quartz tube in the presence of Pt. The sulphur oxides are absorbed on Ag at 450° C and the silver sulphate produced is determined by titration. Cl and Br are determined by burning the substance in oxygen over Ag and absorption in H_2O_2, followed by titration with mercury perchlorate in the presence of diphenylcarbazone as indicator. K. J. GARDNER

1821. **Titrimetric micro-determination of sulphur in compounds containing alkali or alkaline earth [metals].** A. A. Sirotenko (*Mikrochim. Acta*, 1955, [1], 153–154).—When estimating S in metal salts of organic compounds by ignition and subsequent absorption of the SO_3, followed by titration, it was found that some of the SO_3 did not reach the absorber owing to the formation of stable metal sulphates. The S in these was displaced by a second ignition in the presence of 3 to 4 times as much B_2O_3. Both ignitions were carried out in a stream of oxygen in the presence of platinum gauze and the SO_2 was absorbed in 10 per cent. H_2O_2 soln. K. J. GARDNER

1822. **Estimation of sulphur in proteins in general and leather in particular.** P. H. Rao (*J. Amer. Leath. Chem. Ass.*, 1954, **49** [12], 755–757).—Existing methods for the determination of S by oxidation give results that vary according to the oxidising agent used. In the method described, the sample is moistened with water, heated in a nickel crucible, Na_2O_2 is added and heating is continued until the sample is completely oxidised. The crucible is washed with boiling water, then with HCl, and the resulting solution is filtered; the SO_4'' content of the filtrate is determined by pptn. and weighing as $BaSO_4$. Results for S in hide powder tanned with various agents agree well with those given by the Grote - Krekeler method. B. R. HAZEL

1823. **A contribution to the determination of arsenic in organic compounds.** R. Pietsch (*Z. anal. Chem.*, 1955, **144** [5], 353–356).—The method described can be used in the presence of Mg, Ca, Sr, Ba, Co, Ni, Zn, Mn, Cd and Cu and the halogens. *Procedure*—To a 50 to 200-mg sample in a Kjeldahl flask add conc. H_2SO_4 (1 ml), $CuSO_4$ (1 crystal) and conc. HNO_3 (1 ml) and heat to fumes of SO_2. Cool and add further portions of HNO_3 (1 ml) and heat to fuming, until the solution is colourless or pale blue. Transfer the solution to a beaker, dilute to 300 ml, heat to boiling point and add with constant stirring a solution of 7 g of $Ba(NO_3)_2$ in 50 ml of water. Cool, filter and neutralise the filtrate to methyl orange with NaOH soln. Add 25 per cent. aq. HNO_3 (1 drop), run in excess of 0·1 *N* $AgNO_3$ solution and add, dropwise, a conc. solution of sodium acetate, until no more precipitate is formed. Make up to volume, filter and back-titrate the excess of $AgNO_3$ in an aliquot with NH_4CNS soln. 1 ml of 0·1 *N* $AgNO_3 \equiv$ 2·497 mg of As. P. S. STROSS

1824. **Spot test for organic reducing groups with o-dinitrobenzene.** F. Feigl and L. Vokač (*Mikrochim. Acta*, 1955, [1], 101–104).—Organic compounds that act as hydrogen donors in alkaline solution will reduce alkaline ethanolic soln. of *o*- and *p*-dinitrobenzenes, respectively, to the blue - violet or orange salts of quinonoidal nitrogen acids. The reaction with *o*-dinitrobenzene (**I**) is the more sensitive. *Procedure*—Heat one drop of the substance under test with one drop of satd. ethanolic **I** soln. and 0·5 *N* NaOH for up to 1 min.; a blue - violet colour is produced. The limits of detection of 16 compounds were 2·5 to 25 μg. K. J. GARDNER

1825. **Separation and analysis of gases and volatile liquids by gas chromatography.** H. W. Patton, J. S. Lewis and W. I. Kaye (*Anal. Chem.*, 1955, **27** [2], 170–174).—Many gaseous and liquid mixtures (particularly of light hydrocarbons) can be separated and analysed qual. or quant. by gas-chromatographic procedures involving elution by the carrier-gas (H, N or CO_2) from columns (110 cm × 0·5 cm) of charcoal, silica gel or Al_2O_3. A stream of carrier-gas passing continuously through the system causes each component in the sample to move through the column (at 25° to 200° C) at a rate that depends on how strongly it is held by the absorbent. In an efficient separation each component passes from the column into the reference-channel of the detector (a modified thermal conductivity cell) at a different time. The separated fractions can be analysed further by i.r. absorption, mass spectrometry, etc. Choice of carrier and absorbent, construction of sample-container, column and detector, and operation of the entire assembly are described fully, and typical elution curves (detector-response *vs.* time) for different hydrocarbon mixtures are shown. Quant. data are obtained from the elution-peak areas (measured by planimeter or calculated as the product of the peak height and its half width), by means of calibration curves obtained by passing measured vol. of the pure gases through the column. The technique is most suited to non-polar compounds having b.p. < 150° C at atm. pressure; separation of polar compounds is poor unless use is also made of the displacement or gas - liquid partition methods. Hydrogen, methane and ethane

in hydrocarbon gases can be detected at concn. of $\simeq$ 0·01 per cent., C_3 and C_4 hydrocarbons in petrol at concn. of $\simeq$ 0·2 per cent., whilst the quant. determinations are accurate to within $\pm$ 1 to 2 per cent. of the amount found. W. J. BAKER

1826. **The determination of very small quantities of alcohols in aqueous solution at high dilution.** A. von Wacek and F. Zeisler (*Mikrochim. Acta*, 1955, [1], 29–36).—The method is an improvement of that described by Fischer and Schmidt (*Ber.*, 1924, **57**, 693) and enables 50 to 5000 μg of alcohol to be determined in 75 minutes. The solution of alcohol is treated with $NaNO_2$ and acetic acid in a current of purified nitrogen. The highly volatile alkyl nitrites produced are carried over into aq. KI soln. and an equivalent amount of I is liberated, which is titrated against 0·25 *N* sodium thiosulphate. The apparatus is described in detail and operating instructions and tables of results are given. K. J. GARDNER

1827. **Determination of but-2-yne-: 4-diol and other alkynyl alcohols by the Kaufmann solution.** E. Tamate and S. Kinoshita (*J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1954, **57** [12], 922–925).—The determination of but-2-yne-1:4-diol is carried out satisfactorily with Kaufmann soln. (an excess of 100 to 300 per cent.) in an ice-cooled soln. of methanol. In an aq. soln., some substitution of Br takes place and gives a positive error. The influence of formaldehyde and formic acid can be reduced by adding 10 per cent. HCl (2 ml); the positive error due to formaldehyde or H_2O must be corrected by a calibration factor. Propargyl alcohol (prop-2-yn-1-ol) can be similarly determined with a little less Kaufmann soln. (reaction time 60 to 180 min.). The sum of these two alkynyl alcohols can also be obtained similarly, and by subtracting the amount of prop-2-yn-1-ol (obtained by another method, *e.g.*, Nessler's), the amount of butynediol in the reaction mixture can be found. Secondary alkynyl alcohols react with Kaufmann soln., but not tertiary alcohols. K. SAITO

1828. **A note on Eegriwe's test for formaldehyde.** F. Feigl and L. Hainberger (*Mikrochim. Acta*, 1955, [1], 110–111).—The method as described by Eegriwe (*Z. anal. Chem.*, 1937, **110**, 22) is sensitive to 1 in 360,000. If, however, the dry compound is treated with a drop of chromotropic acid in conc. H_2SO_4, a violet coloration is produced in the presence of 1 in 5,000,000 of formaldehyde. K. J. GARDNER

1829. **Column chromatography of sugars.** R. L. Whistler (*Science*, 1954, **120** [3126], 899–900).—Carbon columns should be given a preliminary wash with 1 per cent. HCl followed by dist. water to remove basic ash. Celite, used to improve flow rate, sometimes dissolves in the developing soln. and to avoid its use charcoal is selected which passes a 40- or 60-mesh but is retained by an 80-mesh screen, and used without admixture. Cellulose columns sometimes give rise to the appearance of extraneous carbohydrates; to reduce the amounts of such substances a preliminary wash with water is recommended. Positive pressure should be applied to the head of the column rather than suction on the eluate as the lower portion is liable to become free of solvent, which leads to poor separation. In packing the tubes by the slurry method the slurry must be thick, otherwise fractional separation of particle size occurs. H. F. W. KIRKPATRICK

1830. **Cyclitol series. XX. Paper chromatographs of cyclitols and cycloses.** T. Posternak, D. Reymond and W. Haerdi (*Helv. Chim. Acta*, 1955, **38** [1], 191–194).—The methods that are useful in the paper chromatography of cyclitols and related compounds are described, and figures are given for the R_F values of 33 compounds in butanol - acetic acid - water, *s*-collidine - water, phenol - water and acetone - water. The R_F value rapidly diminishes as the number of hydroxyl groups increases; it is increased by the presence of O-methyl, C-methyl, or C-halogenomethyl groups. All the compounds may be developed by Tollens' phloroglucinol reagent; other reagents are described which selectively develop cycloses, and inositols and inososes. Cyclitols that are susceptible to biochemical oxidation may be detected by treatment with *Acetobacter suboxidans*, followed by one of the cyclose reagents. H. P. PAGET

1831. **Indirect polarographic determination of propylenediamine.** A. D. Horton, P. F. Thomason and M. T. Kelley (*Anal. Chem.*, 1955, **27** [2], 269–271).—Propylenediamine (**I**) is determined indirectly by polarographic determination of the **I** - Cu complex in 0·1 *M* KCl base soln. The propylenediamine sample is added to $CuCl_2$ soln. in which the Cu content is always in stoicheiometric excess of the **I** to be determined. A double wave is obtained: the first is due to excess of Cu and the second to the **I** - Cu complex. The $E_{\frac{1}{2}}$ value for the latter varies between $-0{\cdot}4$ and $-0{\cdot}6$ V *vs.* the S.C.E. according to the **I** concn. of the sample. A linear graph is given by the concn. range 26 to 97 μg per ml, with an overall relative standard deviation of 0·7 per cent.; the limit of detection is 8·8 μg per ml, and the max. concn. that could be determined is 114·4 μg per ml. G. P. COOK

1832. **Direct polarographic determination of aliphatic nitro compounds in presence of lanthanum or cerium salts.** R. Miquel and A. Condylis (*Bull. Soc. Chim. France*, 1955, [2], 236–239).—Polarography of aliphatic nitro compounds in a base electrolyte of H_2SO_4 or a buffer gives waves which decrease considerably with time and do not obey the Ilkovič equation. In a base electrolyte of La acetate (9 g), $BaCl_2$ (11 g) and H_2O (to 1 litre), a wave is obtained which is stable with time but does not give a horizontal step at the end of the wave. The presence of dissolved O is necessary for the stability of the wave. A base electrolyte of $Ce_2(SO_4)_3$ (5 g), H_2SO_4 (3·5 to 4 g) and H_2O (to 1 litre) (more stable than a less acid electrolyte, also satisfactorily used) gives a very stable wave with a horizontal step. Oxygen must not be removed and the wave height includes a value for the reduction of the O. Results for nitromethane, nitroethane and nitropropane are quoted to show the accuracy. Ageing does not affect the height of the wave, but the half-wave potential becomes more negative. There is no max. in the wave even without suppressors and the Ilkovič equation is followed. E. J. H. BIRCH

1833. **Researches related to the analysis of alkyl sulphates and alkylarylsulphonates.** A. Lassieur (*Chim. Anal.*, 1955, **37** [2], 39–44).—The difficulties of qual. and quant. analysis of mixtures of alkyl sulphates and alkylarylsulphonates are stressed. The effect of HCl in the cold or on heating is described with reference to titration of the products of hydrolysis. Dehydration by boiling, detected by increase in iodine value, is sometimes useful for distinguishing secondary and primary alcohols. Attempts to use the phthalic anhydride

derivatives or the varying rates of esterification with acetic acid for analytical purposes were unsuccessful.
A. R. ROGERS

1834. **Infra-red spectra of aromatic compounds. I. The out-of-plane C–H bending vibrations in the region 625 to 900 cm^{-1}.** M. Margoshes and V. A. Fassel (*Spectrochim. Acta*, 1955, **7** [1], 14–24).—The i.r. spectra of a large number of substituted benzenes have been examined to determine the effect of substituents on the out-of-plane hydrogen-bending vibrations between 625 and 900 cm^{-1} (11 to 15 μ). For monosubstituted benzenes of the type C_6H_5M, the frequency of the absorption band between 725 and 775 cm^{-1} decreases linearly with the reduced mass of the diatomic group *M*–carbon. Most monosubstituted benzenes not of this type also show some correlation between frequency and mass of the atom attached to the ring, although the band is shifted to 780 to 820 cm^{-1} when the substituent is —COOH, —COOR (R = ethyl or Na), —COCl, —CCl_3 or —NO_2. Similar variations in frequency (690 to 880 cm^{-1}) with the nature of the substituent have been observed in disubstituted benzenes, and in a few instances of symmetrical trisubstitution. In *p*-disubstituted and in 1:2:4-trisubstituted benzenes, a very strong additional absorption band at $\simeq 13\ \mu$ is present when one of the substituents is a NO_2 or COOH group or an ester or salt of a COOH group. This band is very probably an out-of-plane hydrogen-bending vibration.
W. J. BAKER

1835. **Far ultra-violet absorption spectra of unsaturated and aromatic hydrocarbons.** L. C. Jones, jun., and L. W. Taylor (*Anal. Chem.*, 1955, **27** [2], 228–237).—To determine the analytical possibilities of far-u.v. absorption spectroscopy, the spectra of 69 pure hydrocarbons have been measured, either as vapour or solution, in the range 170 to 230 mμ. The vapour-handling system, the prep. of solvents and solutions, the recording vacuum spectrometer (with 1-metre concave grating and photo-electric detector), and the operational technique are described fully. The far-u.v. spectra of typical paraffins, naphthenes, cyclic and acyclic mono-olefins, allenes, conjugated and non-conjugated diolefins, acetylenes, benzenes and naphthalenes, etc., are shown and discussed generally in relation to mol. structure. For 32 of these compounds, the wavelengths of sharp absorption bands found agree with those given in the literature, but the band intensities are $\simeq$ 12 per cent. higher. The technique is regarded as suitable for analytical purposes and would be of greater value in determining the total concn. of all members of a group of compounds having similar electronic structure than for identifying the separate members of the group. This identification, however, can often be effected from the vapour spectra of many compounds which show numerous sharp Rydberg lines mainly at wavelengths of 100 to 170 mμ.
W. J. BAKER

1836. **Rapid quantitative analysis of anthracene in carbazole or anthraquinone.** E. Funakubo, Y. Matsumoto and T. Kishikawa (*J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1954, **57** [12], 919–922).—Four methods for the determination of anthracene (< 10 per cent.) in carbazole are compared. Neither the oxidation method (quinone method) nor the "xylene distillation method" is satisfactory; the "chlorobenzene partition titration method" is the most satisfactory. *Procedure*—Maleic anhydride (W_m g, $\simeq$ 0·1 g, purity y_1) is added to the sample (W_s g, $\simeq$ 0·5 g) in chlorobenzene (10 ml) and set aside for 2 hr. to complete the reaction. Water (50 to 60 ml) is added and the excess of maleic acid is titrated with 0·1 *N* NaOH soln. (*v* ml) in the presence of the chlorobenzene layer. The anthracene content (*A*) is calculated as follows: A (per cent.) $= 182{\cdot}2(W_m y_1 - 0{\cdot}00491v)/W_s$. When the benzene soln. of the carbazole sample is submitted to chromatography (Al_2O_3 column, $\simeq$ 100 g), only anthracene and phenanthrene pass through the column. Both are weighed together after the removal of benzene from the eluate and the amount of anthracene can be estimated by comparing the m.p. of the mixture with those of a standard series of known compositions, but the accuracy is inferior to that of the titration method. Minor impurities are detected on the column by illuminating it with u.v. light.
K. SAITO

1837. **Rapid quantitative analysis of anthracene. VII.** E. Funakubo, Y. Matsumoto, M. Kawanishi and G. Kon (*J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1954, **57** [12], 916–919).—The accuracy of two methods for the rapid determination of anthracene by use of the addition reaction with maleic anhydride, proposed by the same authors (*Ibid.*, 1954, **57**, 562, 556), is examined and the average deviation for both calculated. Both are superior to the conventional quinone method (oxidation method) in many respects. The "chlorobenzene partition titration method" (average deviation 0·28 per cent. for samples of purity > 50 per cent.; 0·76 for less pure samples) is simpler than the "xylene distillation method" (0·30 for > 50 per cent.; 1·31 for less pure). The errors due to the indicator and CO_2 are negligible for both.
K. SAITO

1838. **Determination of benzo[*a*]pyrene in complex mixtures. Use of catalytic iodination on activated alumina.** R. Tye, M. J. Graf and A. W. Horton (*Anal. Chem.*, 1955, **27** [2], 248–253).—Benzo[*a*]pyrene (**I**) concentrate was obtained from high-boiling petroleum oil by fractional distillation followed by chromatography on alumina columns, the **I**-enriched fractions being selected spectrophotometrically. Two equal portions of the fractions are taken: one is subjected to a catalytic iodination on a column of activated alumina, then both treated and untreated fractions are measured spectrophotometrically. The difference in absorbance gives a measure of the **I** content. A reproducibility of $\pm$ 20 per cent. was obtained at the $\simeq$ 0·01 per cent. concn. level, $\pm$ 10 per cent. at the $\simeq$ 0·10 per cent. level and $\pm$ 5 per cent. for levels > 0·5 per cent. The limit of detection is 0·001 mg. Some polycyclic compounds, in particular alkylbenzopyrenes, are expected to interfere.
G. P. COOK

1839. **Chromatographic separation of phenol and substituted phenols by ion-exchange resin.** T. Seki (*J. Chem. Soc. Japan, Pure Chem. Sect.*, 1954, **75** [12], 1297–1299).—Adsorption-chromatographic distribution of phenol, *p*-cresol and 4-*tert*.-butylphenol is studied with several kinds of cation-exchange resin. Amberlite IR-120 and IRC-50 (either hydrogen form or sodium form) can be used for their separation; the best eluting medium consists of 20 per cent. of *n*-propanol and 80 per cent. of 0·3 *N* HCl. Trinitrophenol, 2:6-dinitrophenol, 2:4-dinitrophenol and *o*-, *m*- and *p*-nitrophenols can be eluted fractionally with an acetate buffer soln. of pH 4·5 from an Amberlite IR-112 column. These results may be utilised for the analysis of substituted phenols after nitration of the phenol.
K. SAITO

1840. **Bromimetric determination of phenols in aqueous solution.** L. von Erichsen and N. Rudolphi (*Erdöl u. Kohle*, 1955, **8** [1], 16–19).—Phenol and eleven homologues have been brominated individually in a flask sealed by aq. KI so that escape of Br was prevented and the reaction could be stopped after a definite time. The phenols fall into the three following groups: (*a*) *m*-cresol, phenol, 3:5-diethyl-, 5-ethyl-3-methyl- and 3:5-dimethylphenol each substitute three atoms of Br per mol. and no more; the last-named reacts more slowly than the others; (*b*) *o*-cresol, 2:3- and 2:5-dimethylphenol each substitute two atoms of Br quickly (in $\simeq$ 30 sec. at room temp.), after which the reaction proceeds very slowly; (*c*) *p*-cresol, 2:4-, 3:4- and 2:6-dimethylphenols, 2:4:5- and 2:3:5-trimethylphenols, and *p*-ethylphenol react at a diminishing rate with no definite stages. The bromine number is thus of analytical value only for mixtures of members of the first two groups.

A. R. PEARSON

1841. **Differentiation of naphthol- and naphthylamine-sulphonic acids by spot tests.** F. Feigl and L. Hainberger (*Mikrochim. Acta*, 1955, [1], 112–117).—Twelve naphthol- (**I**) and naphthylamine-sulphonic acids (**II**) were warmed with a few mg of hexamine and 1 to 2 drops of conc. H_2SO_4. All except one (2-naphthol-6;8-disulphonic acid) of the **I** series gave a violet to brown coloration; the **II** series remained colourless. The test is proposed for differentiating **I** from **II**. The reaction is most sensitive in the total absence of water. The limits of detection are 0·1 to 3 μg. **II** can be detected by adding one drop to one drop of saturated *p*-dimethylaminobenzaldehyde in glacial acetic acid. An orange or yellow colour is produced. The limits of detection are 0·1 to 0·5 μg.

K. J. GARDNER

1842. **Titration of phenolic esters in ethylenediamine.** R. A. Glenn and J. T. Peake (*Anal. Chem.*, 1955, **27** [2], 205–209).—Phenolic esters were determined by acid-base titration in ethylenediamine solution. Three procedures are described: (*a*) titration with potassium methoxide in the presence of *o*-nitroaniline as indicator; (*b*) titration with potassium methoxide, with azo violet as indicator; (*c*) potentiometric titration with glass-platinum electrodes. Recoveries were > 99 per cent. by (*a*) with nine different phenolic esters, and a precision of $\simeq \pm$ 1·5 per cent. was obtained by the potentiometric procedure on 11 different phenolic esters.

G. P. COOK

1843. **Determination of β-ionone in the presence of the α-isomer.** M. Pesez (*Bull. Soc. Chim. France*, 1955, [2], 190).—Although both α- and β-ionone give a red colour with HCl, a mixture of acetic acid and HCl gives this colour with the β-isomer only. *Procedure*—To a solution of $\simeq$ 0·1 g of ionone in 10 ml of acetic acid are added 5 ml of a mixture of 10 ml of conc. HCl with 90 ml of acetic acid, and the mixture is heated at 40° C for 1 hr. After cooling to room temp. in 10 min., the absorption is measured at 550 mμ. Beer's law is followed for amounts of β-ionone up to 5 mg.

E. J. H. BIRCH

1844. **Paper chromatography of cinnamic acid derivatives.** A. H. Williams (*Chem. & Ind.*, 1955, [5], 120–121).—Chlorogenic acid (3-caffeoylquinic acid) gives a single spot on paper chromatography in alcoholic and phenolic solvents, but gives two spots in 2 per cent. acetic acid. The main spot corresponds to that reported by other workers; the second spot of higher R_F is much weaker. A sample of caffeic acid (made by hydrolysis of chlorogenic acid) and all related hydroxycinnamic acids and esters examined, except *o*-coumaric acid, showed the same effect; corresponding hydroxybenzoic acids all gave single spots. The two spots are due to the presence of both *cis*- and *trans*-isomers. Irradiation with u.v. light before chromatography causes an increase in the relative strength of the higher R_F spot.

A. R. ROGERS

1845. **Non-aqueous titration method for determination of the purity of hexahydro-1:3:5-trinitro-*s*-triazine and its content in wax and poly*iso*butylene motor oil compositions.** S. M. Kaye (*Anal. Chem.*, 1955, **27** [2], 292–294).—Hexahydro-1:3:5-trinitro-*s*-triazine (**I**) is determined by titration in dimethylformamide with standard Na methoxide in benzene-methanol, with azo violet as indicator. When applied to purified **I** samples and synthetic samples containing wax and poly*iso*butylene motor oil binder, the method gave an average error of 0·13 per cent. and an average standard deviation of 0·13 per cent.

G. P. COOK

1846. **Infra-red method for determination of *o*-, *m*- and *p*-nitrotoluene and 2:4-dinitrotoluene in mixtures.** F. Pristera and M. Halik (*Anal. Chem.*, 1955, **27** [2], 217–222).—The method involves measurement of a 20 per cent. soln. of the sample in *cyclo*hexane at $\simeq$ 8·70 μ for *o*-nitrotoluene, 12·49 μ for *m*-nitrotoluene, 8·49 μ for *p*-nitrotoluene and 9·39 μ for 2:4-dinitrotoluene. The method also includes the determination of 2:6-dinitrotoluene by measurement of a 25 per cent. benzene soln. at 11·22 μ. Concn. of the constituents are determined by reference to previously established absorptivities. When an ingredient of the sample is > 2 or 3 per cent., the relative error is generally within $\simeq \pm$ 5 per cent., but where the concn. is $\simeq$ 2 per cent. or less the error is sometimes high, in one case being + 38·7 per cent. for a *p*-nitrotoluene determination.

G. P. COOK

1847. **Absorption in the visible and near ultraviolet of some aryl- and aroyl-hydrazones. I. *o*-, *m*- and *p*-Nitrophenylhydrazones.** P. Grammaticakis (*Bull. Soc. Chim. France*, 1954, [11-12], 1372–1380).—Observations are made in the region 220 to 600 mμ of the absorption spectra in alcoholic solution of *o*-, *m*- and *p*-nitrophenylhydrazines, and of the corresponding nitrophenylhydrazones derived from acetone, *cyclo*hexanone, acetophenone, benzophenone, propionaldehyde, crotonaldehyde, benzaldehyde, cinnamaldehyde, furfuraldehyde, *o*-, *m*- and *p*-hydroxybenzaldehydes, *o*-, *m*- and *p*-methoxybenzaldehydes, and *o*-, *m*- and *p*-nitrobenzaldehydes. The bands are classified into "nitro" bands and "phenylhydrazone" bands; the qual. relationships observed between the spectra of a set of phenylhydrazones are similar to those between the spectra of the corresponding derivatives of a given nitrophenylhydrazine. The qual. relationships between the spectra of the three nitrophenylhydrazones of a given carbonyl compound are similar to those between the spectra of the three nitroanilines or the three nitrophenylhydrazines. The compounds studied are briefly characterised.

H. P. PAGET

1848. **Absorption in the visible and near ultraviolet of some aryl- and aroyl-hydrazones. II. *o*-, *m*- and *p*-Carboxyphenylhydrazones.** P. Grammaticakis (*Bull. Soc. Chim. France*, 1954, [11–12],

1381–1390).—Observations similar to those described in Part I (*Anal. Abstr.*, 1955, **2**, 1847) are given for *o*-, *m*- and *p*-carboxyphenylhydrazines, and for the corresponding carboxyphenylhydrazones of the compounds listed in Part I. Carboxylation of phenylhydrazones results in small changes in the strongest bands without alteration of the main qualitative relationships between them; the variations produced in the absorption curves are more significant for the phenylhydrazones of acetone and specially related compounds than for the phenylhydrazones of aromatic aldehydes and ketones.
H. P. PAGET

1849. **Absorption in the visible and near ultraviolet of some aryl- and aroyl-hydrazones. III. Hydroxybenzoylhydrazones.** P. Grammaticakis (*Bull. Soc. Chim. France*, 1954, [11–12], 1391–1397).—Observations similar to those described in Part I (*Anal. Abstr.*, 1955, **2**, 1847) are made on *o*- and *p*-hydroxybenzoylhydrazines, and on the corresponding hydroxybenzoylhydrazones of some of the compounds listed in Part I. The spectrum of *o*-hydroxybenzoylhydrazine is very similar to that of *o*-hydroxybenzamide, but differs considerably from that of benzoylhydrazine. Ortho-hydroxylation of the benzoylhydrazones has a significant effect only with acetone and compounds spectrally related to it, the qualitative spectral relationships of the other compounds being unchanged. The spectrum of *p*-hydroxybenzoylhydrazine is very similar to that of *p*-hydroxybenzamide; para-hydroxylation of the benzoylhydrazones has very little influence on their spectra.
H. P. PAGET

1850. **Determination of hydrocarbons in hydrogen by a palladium tube / mass spectrometer method.** R. A. Brown, H. B. Ogburn, F. W. Melpolder and W. S. Young (*Anal. Chem.*, 1955, **27** [2], 237–239).—An apparatus and method for concentrating hydrocarbons in hydrogen-rich samples by pumping hydrogen through a palladium tube kept at $\simeq$ 500° C and containing the sulphur- and oxygen-free sample ($\simeq$ 125 ml) is described. The concentrate can be obtained in $\simeq$ 20 min. The assembly comprises an oxygen analyser, sample reservoir, palladium-tube section, and means for transfer of residual gas to a mass spectrometer in which the hydrocarbons are analysed separately. From 0 to 3 per cent. of hydrocarbons in the C_1 to C_7 range can be determined with an error of 0·1 per cent. and a reproducibility of 0·05 per cent. The composition of the original sample is calculated from the vol. of hydrogen pumped away and the composition and vol. ($\simeq$ 50 ml) of the hydrocarbon concentrate. Results for several hydrogen - hydrocarbon mixtures and for the determination of $\simeq$ 0·2 mol. per cent. of *n*-hexane in a synthetic mixture are reported and discussed briefly in relation to the validity of the method.
W. J. BAKER

1851. **Application of separation techniques to a high-boiling shale-oil distillate.** G. U. Dinneen, J. R. Smith, R. A. Van Meter, C. S. Allbright and W. R. Anthoney (*Anal. Chem.*, 1955, **27** [2], 185–190).—The fractionation of a gas oil, having a boiling range 625° to 1100° F, from Colorado shale was studied by using adsorption, vacuum distillation, thermal diffusion and complex-formation techniques. The oil, a complex mixture of saturated, olefinic and aromatic hydrocarbons, together with O-, N- and S-containing compounds, was progressively fractionated to give high-quality concentrates of the important groups of compounds. These were further separated into simple fractions; the concentrates containing the saturated and olefinic materials were further subdivided into straight- and branched-chain compounds, and aromatic fractions were subdivided according to the number of condensed aromatic rings. The N concentrates were separated into fractions containing different relative amounts of basic and non-basic nitrogen.
G. P. COOK

1852. **Chromatographic determination of the chemical group composition of the middle fractions of shale tar.** A. Ya. Aarna and K. A. Kask (*Tr. Tallinskogo Politekhn. In-ta*, 1953, [51], 1–15; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 40,038).—A chromatographic method for separating the middle fractions of shale tar (boiling range 200° to 320° C) on silica gel, without the use of a solvent, is described; butanol is used as displacer. The ratio of paraffin to naphthene hydrocarbons is determined from equations relating refractivity and boiling point, and the ratio of naphthenes to unsaturated hydrocarbons from bromine number and mol.-wt. measurements. Saturated hydrocarbons can be separated from unsaturated, and unsaturated from aromatic, but the separation of aromatic hydrocarbons from oxygen and sulphur compounds is incomplete.
E. HAYES

1853. **Determination of sulphur in volatile hydrocarbon mixtures by a lamp-conductimetric method.** E. R. Quiram (*Anal. Chem.*, 1955, **27** [2], 274–277).—A simple and rapid conductimetric method of determining any concn. of S in volatile hydrocarbon mixtures burnt from a wick-fed lamp is described fully. The all-glass absorption assembly enables measurements to be made in one container, and errors due to temp. changes and dissolved CO_2 are eliminated. The lamp chimney and attached bubbler are connected directly to the absorption flask (a modified 50-ml graduated cylinder) containing 20 ml of freshly prepared 3 per cent. H_2O_2, and provided with hooks on which to fit the conductivity cell. Combustion of the sample (3 to 5 g) in an atm. of 65 per cent. of CO_2 and 35 per cent. of O is continued for $\simeq$ 1 hr.; the absorption soln. (made up to 25 ml) is aerated with purified air for 5 min., the conductivity cell is inserted and the reading recorded at 25° C. The wt. of sample and of absorption soln. in the flask after burning are obtained by successive weighings of the lamp and absorption assemblies. The calibration curve is determined from the measured specific conductances of aq. 0·1249 *N* H_2SO_4 (1 ml $\equiv$ 2 mg of S) and of more dil. solutions prepared therefrom. Eight determinations of S can be made in about three hr. with eight separate assemblies; the procedure is regarded as an improvement over the turbidimetric, gravimetric and volumetric methods, but aldehydes, halogens, nitrogen and other acid-forming substances interfere.
W. J. BAKER

1854. **Separation of sulphur compounds from petroleum.** C. J. Thompson, H. J. Coleman, H. T. Rall and H. M. Smith (*Anal. Chem.*, 1955, **27** [2], 175–185).—Physical and chemical methods for the separation of sulphur from petroleum were investigated, and include distillation, adsorption and thermal diffusion techniques. Distillation techniques can be used only under carefully controlled conditions of temp., heating time and metal contact to eliminate thermal decomposition. The most effective separation was achieved by chromatography on alumina, and thermal diffusion techniques followed by refractive index, i.r. or mass-spectra measurements on the fractions showed

many advantages over conventional methods. Chemical methods were more useful in separating different classes of S compounds from each other after initial separation from hydrocarbons. A list of 43 sulphur compounds found in a crude oil is given. G. P. Cook

1855. **Coulometric titration of dyes with externally generated titanous ion.** J. S. Parsons and W. Seaman (*Anal. Chem.*, 1955, **27** [2], 210–212).—The method is based on the generation of $TiCl_3$ externally at a mercury-pool cathode, which has a current efficiency of 99·8 per cent. Successful titration of Orange II, tartrazine, *p*-aminoazobenzene, amaranth and methyl violet was achieved, giving a standard deviation of $\pm$ 0·15 per cent. Results were in satisfactory agreement with those from the conventional $TiCl_3$ method. An indirect method, in which an excess of 0·4 *N* $TiCl_3$ is added and the dye determined by back-titration with 0·025 *N* amaranth, is used for oil-soluble dyes. G. P. Cook

1856. **The polarographic reduction of amaranth.** G. G. McKeown and J. L. Thompson (*Canad. J. Chem.*, 1954, **32** [11], 1025–1032).—Amaranth, an azo dyestuff used in food colouring, has been studied polarographically between pH 2·0 to 10·0 in aqueous solutions. The diffusion current varied with the concn., was independent of pH and free from excessive maxima distortion. A concn. of 8·3 μM gives a current of $\simeq$ 0·1 μA. Since $E_{\frac{1}{2}}$ varies by — 0·080 V per pH unit, and logarithmically with concn., attention must be paid to these variants if the qual. and quant. determinations are to be of any value. D. G. Higgs

1857. **Studies in coal-tar colours. XV. FD & C Red No. 1, D & C Red No. 5 and some related colours.** L. S. Harrow and J. O. Millham (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 172–182).—The spectrophotometric properties of FD & C Red No. 1 [Ponceau 3R, 1-(3:4:6-trimethylphenylazo)-2-naphthol-3:6-disodium sulphonate] and D & C Red No. 5 [Ponceau 2R, 1-(2:4-dimethylphenylazo)-2-naphthol-3:6-disodium sulphonate] have been investigated, together with the properties of likely contaminants. Solutions of the dyes in 0·02 *M* ammonium acetate conform to Beer's law. A. A. Eldridge

1858. **Improvement of the Kürschner and Hoffer method for cellulose determination by addition of sulphuric acid to the main reagent.** M. Krajčinovič, R. Račev and F. Paro (*Papier. Darmstadt*, 1954, **8** [21–22], 482–484).—Experimental results are tabulated to show that the Kürschner and Hoffer method for the determination of cellulose can be improved by the addition of 0·8 g of H_2SO_4 (sp. gr. 1·84) to the main reagent, which consists of 20 ml of ethanol (96 per cent.) and 5 ml of HNO_3 (sp. gr. 1·41). By using this modified reagent, the determination can be carried out more quickly and accurately; the cellulose obtained is pure and its oxidative degradation by the action of the HNO_3 present in the reagent is reduced. The pentosan content in the residual cellulose is slightly lower. Brit. Cotton Ind. Res. Ass. Abstr.

1859. **Measurement of retention of locust-bean gum [in paper] by analysis of sugar units.** H. S. Leech (*Tappi*, 1954, **37** [7], 290–294).—A new method described has the advantage that it can be used for testing prepared paper samples and therefore measures the true gum content of the paper. The steps involved are hydrolysis with dilute sulphurous acid, recovery of sugars, separation of the sugars by paper partition chromatography and determination of the sugars with sodium periodate. Brit. Cotton Ind. Res. Ass. Abstr.

1860. **Semi-micro estimation of urea in treated paper.** A. L. Glass (*Chemist Analyst*, 1954, **43** [4], 104).—The standard clinical procedure for the determination of urea has been adapted for use with urea-treated paper. *Procedure*—3 sq. in. of paper are extracted with a suitable volume of H_2O (15 ml for 0·5 g per sq. ft.). The soln. is run into sodium hypobromite soln. (1 ml of Br added to 40 ml of aq. 20 per cent. NaOH immediately before use) in a semi-micro ureometer (described). The recovery is 97 per cent. and the method can be used with papers treated with nitrate and a salt of an amino acid. G. B. Thackray

1861. **Identification of the colouring matter of cosmetics by paper chromatography.** J. Deshusses and P. Desbaumes (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [6], 484–489).—Paper chromatography was used to separate and identify the colouring matter of the following cosmetics: tinting solutions or shampoos and other hair-dyeing products. The colouring matter of cosmetics is so varied that a solvent to separate all of them has not been found, but 1 per cent. ammonia solution saturated with butanol permits the separation of trinitrophenol and nitro-*o*-phenylenediamine, in which solvent nitro-*p*-phenylenediamine gives a reddish coloration. R. J. Magee

1862. **Identification and determination of thioglycollic acid in cold permanent-wave preparations.** H. Forster, A. Meyer and H. Volkart (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [6], 490–495).—The identification of thioglycollic acid in cold-wave preparations is described. This can be carried out by means of the colour reaction with sodium nitroprusside or ferric chloride in acid and alkaline media, or by the precipitation reaction with a cadmium salt, according to the method of Kramer (*Brit. Abstr. C*, 1953, 165). The following two methods are used for the quantitative determination of the thioglycollic acid: (*a*) direct iodimetric titration combined with a sulphite determination and (*b*) iodimetric titration of the cadmium precipitate. Results for twenty-six cold-wave preparations examined by both methods gave results which agreed closely. R. J. Magee

1863. **Separation of polyhydric alcohols from alkyd resins and their identification.** C. P. A. Kappelmeier (*Verfkroniek*, 1954, **27** [11], 291).—Alkyd resins undergo aminolysis on treatment with NH_3 or an amine, the acids are converted into amides (insoluble in H_2O) and the alcohols are liberated (generally soluble in H_2O). As a qualitative test for pentaerythritol (**I**) in alkyds, 3 g of the resin are refluxed with 6 ml of 2-phenylethylamine for 3 hr. Pure **I** is recovered by washing the product with chloroform to remove the amides and any glycerol present. In the presence of glycerol, the yield of **I** is $\simeq$ 2 per cent. low owing to the solubility of **I** in glycerol. Other solvents tested did not give better results. Rosin and maleic esters resist aminolysis by this procedure, even when triethylenetetramine is used, and the method cannot be used for their analysis. D. R. Duncan

1864. **Identification of epoxy resins.** G. Lewin, H. Gerlings and A. Wijling (*Verfkroniek*, 1954, **27** [11], 292; translation in *Paint Manuf.*, 1954, **24**,

434).—If the resin is available (not in a varnish or paint medium), 0·5 to 1 g is boiled for 3 min. with a solution of 30 mg of *p*-phenylenediamine in ≃ 8 ml of distilled H_2O. In the presence of an epoxy resin (**I**) a pink colour develops in the aqueous layer, increasing in intensity on being set aside for 12 hr. If a varnish or paint medium, which may contain oils and alkyds but is free from phenolic and nitrogenous resins, is to be tested, the sample is dissolved in Cellosolve (ethoxyethanol) and saponified with alcoholic *N* KOH. **I** is pptd. quant. on the addition of H_2O and the other products are dissolved. **I** may be identified as above; it gives a bright orange colour in the Echtrotsalz test (Kappelmeier, *Farben-Ztg.*, 1937, **42**, 561), but phenolic resins interfere, giving a deep-red coloration. In the presence of phenolic, urea and melamine resins, acid hydrolysis under the conditions specified by Kappelmeier (*Chem. Weekbl.*, 1937, **34** [12], 217) should be employed. The product is extracted with ether to remove the products derived from oils and alkyd resins; the epoxy resin can then be extracted from the residue by means of Cellosolve. D. R. DUNCAN

1865. **Ultra-violet spectrophotometric determination of polymerised styrene in styrenated fatty acids and alkyd resins.** R. C. Hirt, R. W. Stafford, F. T. King and R. G. Schmitt (*Anal. Chem.*, 1955, **27** [2], 226–228).—Polymerised styrene is determined in the presence of monomeric styrene and conjugated triene acids by u.v. absorption at 291, 282 and 269 mμ, the last being the analytical wavelength for polystyrene; the absorbancy at the other wavelengths enables correction for interference by monomeric styrene and the triene acids at the analytical wavelength. Results on styrenated fatty acids obtained by this method were more accurate than those from the chemical method based on saponification. The application to the determination of polystyrene in alkyd resins is also described.
G. P. COOK

1866. **New possibilities in infra-red spectrometry of varnishes.** H. G. Dietz (*Peint.-Pigm.-Vern.*, 1954, **30**, 820–822).—The potassium bromide compressed-pellet technique enables materials that are insoluble in spectroscopic solvents to be examined. Cured thermosetting resins may be identified by spectra obtained in this way and some typical spectra are shown. An approx. estimate of the proportion of dipentaerythritol in pentaerythritol may be obtained by the KBr technique based on the CH vibration doublet at 3·4 μ. L. A. O'NEILL

1867. **Determination of natural rubber by titration with permanganate in non-aqueous media.** J. Chrastil (*Chem. Listy*, 1954, **48** [5], 781–783).—A rapid procedure is described for the volumetric determination of natural rubber extracted from plant material. Dried roots and leaves of koksaghyz (*Taraxacum kok-saghyz*) (1 to 5 g) are submitted to a preliminary extraction for 10 hr. with acetone (50 ml) to remove resinous matter, followed by extraction with 3 portions of CCl_4 (for 3 × 5 hr.) in a special apparatus (illustrated). The combined CCl_4 extracts are diluted to 100 ml, an aliquot (1 to 5 ml) is treated with a (1 + 1) mixture of 96 per cent. H_2SO_4 and glacial acetic acid (1 to 5 drops), and the soln. is titrated with a 0·01 *N* soln. of $KMnO_4$ in glacial acetic acid until a pink colour persists for 10 min. One isoprene unit is equivalent to one atom of oxygen. A blank is subtracted.
G. GLASER

See also Abstract 1748.

4.—BIOCHEMISTRY

INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

1868. **The manometric analysis of respiratory gases in blood containing volatile anaesthetic agents.** D. A. Holaday and M. Verosky (*J. Lab. Clin. Med.*, 1955, **45** [1], 149–152).—From a comparative study of three methods, the one described by Goldstein *et al.* (*Brit. Abstr. C*, 1950, 283) is found to be the most satisfactory for the combined determination of O and CO_2 in blood containing any of the volatile anaesthetics, and it is recommended for use irrespective of whether or not such anaesthetics are present. W. H. C. SHAW

1869. **Micro-chromatography. II. Detection of trace elements in biological media.** K. Lakshminarayanan (*Proc. Indian Acad. Sci.*, 1954, **40 B** [6], 167–172).—A simple method is described for the separation of 0·15 to 0·2-μg quantities of Cu, Co, Ni, Fe, Mn, Al, B, Mo and Zn in 0·25 μl of different media. The solution is spotted on to a filter-paper disc and developed for 40 to 45 min., the diameter of the solvent boundary being 2·3 cm; the paper is subsequently air-dried and sprayed with detecting agents. Solvent mixtures employed are *n*-butanol - acetic acid - water (4:1:5), aq. lutidine and aq. collidine. Some R_F values are given.
G. R. WHALLEY

1870. **An accurate micro-method for the estimation of magnesium in biological material, particularly blood serum.** K. J. Karrman and S. Borgström (*Svensk Kem. Tidskr.*, 1955, **67** [1], 18–23).—Serum (1 ml) or other solution is dried and ignited at 700° to 800° C for 10 min. When cool, *N* HCl (0·5 ml) is added and the contents of the crucible are washed with water into a test-tube to a total vol. of about 4 ml. Next, conc. aq. NH_3 (0·5 ml), sodium oxalate soln. (1 ml) and water to a total vol. of about 8 ml are added to each tube, which is then heated on a water bath for 2 hr. The calcium oxalate is filtered off by means of a filter needle with a cotton-wool filter (according to Pregl) and washed with 3 × 0·5 ml of water. The filtrate is then titrated from an Agla micro-burette with disodium ethylenediaminetetra-acetate (0·00252 *M*) with a solution of Eriochrome black T as indicator. Tests in the presence of organic matter, phosphate, Ca, Mn, Fe, S and SiO_2 showed no interference, the error being from − 1·5 to + 1·25 per cent. N.E.

1871. **Estimation of serum copper with sodium diethyldithiocarbamate.** R. J. H. Scholtis (*Nederland. Tijdschr. Geneesk.*, 1954, **98**, 1795–1798).—The method is an extension of that described for estimation of Fe with phenanthroline (*Ibid.*, 1954, **98**, 1636). Serum is diluted and acidified with HCl. Protein is pptd. with trichloroacetic acid and removed. Fe is eliminated with citric acid, a 50 per cent. alcoholic solution of ammonia is added and Cu is estimated with Na diethyldithiocarbamate soln. at 430 mμ in a photo-electric colorimeter. The preparation of the necessary calibration curve is described. Both Fe and Cu can be estimated in 6 ml of serum. NUTR. ABSTR. AND REV.

1872. **Micro-determination of lead in blood by polarography.** R. Jensen (*Chim. Anal.*, 1955, **37** [2], 53–55).—Blood (5 g) is ashed at 500° C with 50 per cent. $Mg(NO_3)_2$. The residue is treated with 50 per cent. HNO_3, converted into oxides at 500° C,

then gently heated with fuming HCl. Any $Fe^{\cdots}$ (which would interfere) is reduced to $Fe^{\cdot\cdot}$ by gently boiling with an acid soln. of hydroxylamine hydrochloride. The pH is reduced to about 1·0 with conc. HCl, carboxymethylcellulose is added to inhibit maxima, and the soln. is examined polarographically from — 0·3 to — 0·6 V. Recovery and blank determinations are advised. The error is $\simeq \pm 10$ per cent. A. R. ROGERS

1873. **Blood iodine determination with radioactive iodine (preliminary communication).** H. Spitzy (*Mikrochim. Acta*, 1955, [1], 130–133).—Klein (*Anal. Abstr.*, 1955, **2**, 700) describes a method of estimating blood iodine in which the catalytic action of iodine on the discoloration of ceric sulphate by arsenous acid is used. Although extremely sensitive, this method gives unreliable results. The various stages were checked with ^{131}I. It was found that the losses occurred mainly during the distillation of the HI. K. J. GARDNER

1874. **Polarographic determination of ethanol in blood.** D. Monnier and W. F. Rüedi (*Helv. Chim. Acta*, 1955, **38** [2], 402–410).—The determination of ethanol in blood, in the presence of up to 6 per cent. of acetone, is described. *Procedure*—Blood (5·0 ± 0·05 g) is distilled with 6·5 times its volume of saturated aq. trinitrophenol (to prevent frothing). The ethanol is converted into acetic acid by HNO_3 - $K_2Cr_2O_7$ [2·20 g of $K_2Cr_2O_7$ in 250 ml of HNO_3-free HNO_3 (sp. gr. 1·40)]. The reaction is stoicheiometric under the experimental conditions, when 4·26 mg of $K_2Cr_2O_7 \equiv 1$ mg of ethanol. The approx. ethanol content should be known, so that only a slight excess of $K_2Cr_2O_7$ is added; this content is ascertained by adding to 5 ml of the distillate 3 to 5 ml of conc. HNO_3 and 1 ml of reaction mixture. If a yellowish colour persists after the solution has been heated for 1 min., then an excess of $K_2Cr_2O_7$ is present, but, if the colour is blue - green, it indicates an excess of ethanol, and repeated 1-ml quantities of reaction mixture are added until the colour is yellow. When the ethanol content is > 3 per cent., the solutions are diluted. A 5-ml aliquot is treated with 1, 2 or 3 ml of acid dichromate as previously determined and heated for 5 min. on a steam-bath. When cold, 2 ml of the oxidised soln. are treated with 4 ml of 4 *N* NaOH, to give a final alkalinity of approx. 1 *M*. The soln. is deoxygenated by a stream of pure H or N and then polarographed ($E_{\frac{1}{2}} = -0{\cdot}85$); the effect of O is, however, negligible. Under the conditions described the max. error rarely exceeds 2 per cent. for ethanol contents between 0·5 and 3 per cent. in the presence of up to 6 per cent. of acetone. D. G. HIGGS

1875. **Critical study of the determination of alcohol in blood.** P.-E. Wenger, D. Monnier and W. F. Rüedi (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [6], 528–552).—A systematic study of the different methods employed in Switzerland for the determination of alcohol in blood, with particular reference to the magnitude of the errors and reproducibility of results, is undertaken. The following methods are examined in detail: (*i*) determination according to Nicloux (direct method), (*ii*) determination according to Rochat (*Helv. Chim. Acta*, 1946, **29**, 102, 819), (*iii*) determination according to Schifferti (*Praxis*, 1949, p. 22; and 1951, p. 34) and (*iv*) determination in expired air according to Haarger (ethylometer). None of these methods is entirely satisfactory. Results should be given with the precision of the method used. R. J. MAGEE

1876. **Separation of α-keto acid dinitrophenylhydrazones by paper electrophoresis and their colorimetric determination.** H. Tauber (*Anal. Chem.*, 1955, **27** [2], 287–289).—A method is described for the determination of pyruvic acid and α-oxoglutaric acid. The acids are pptd. as the 2:4-dinitrophenylhydrazones which are then separated by paper electrophoresis. The separated derivatives are dissolved in *N* NaOH and are determined colorimetrically, with a green filter. Application of the method to mammalian blood yielded recoveries between 88 and 110 per cent. at the 200 to 300-μg level in 10 ml of blood for pyruvic acid and 81 to 85 per cent. at the 50 to 100-μg level for α-oxoglutaric acid. G. P. COOK

1877. **The influence of amino acids on the determination of reducing carbohydrates.** H. Iwainsky (*Z. LebensmittUntersuch.*, 1955, **100** [3], 173–179).—The presence of amino acids, which are not removable by the usual clearing agents, interferes appreciably with the accuracy of sugar determinations by the Luff - Schoorl, Bleyer - Sichert and Hagedorn - Jensen methods. Results by the Meissl - Allihn method are slightly increased by the presence of glycine. The chromatographic anthrone method of Dimler, Schaefer, Wise and Rist (*Brit. Abstr. C*, 1953, 11) is free from these objections for 5 amino acids (not including tryptophan). P. S. ARUP

1878. **The estimation of heparin and similar substances in human blood and tissues using a combined biological and colorimetric method with paper-electrophoretic studies.** M. Bassiouni (*J. Clin. Pathol.*, 1954, **7** [4], 330–335).—A method for extracting heparin from blood and tissues is described: the extract is purified by treating the complex formed with the dye Azure A with formamide, followed by pptn. with methanol in the presence of NaCl. The biological evaluation of active heparin is based upon the observation that within a certain range of concn. of heparin added to re-calcified sheep plasma, the log. of the clotting time is proportional to the amount of heparin added. The colorimetric method consists in pptn. of the heparin with Azure A, washing and re-dissolving in a buffered solvent mixture, followed by absorptiometric estimation of the dye. Standard curves are prepared for each method. H. F. W. KIRKPATRICK

1879. **The estimation of Butazolidin [phenylbutazone] in blood.** D. G. Moss (*J. Clin. Pathol.*, 1954, **7** [4], 344–347).—Add 1 ml of serum to 4 ml of acetone in a glass-stoppered centrifuge tube, shake, set aside for 30 min. and centrifuge. Transfer 1-ml duplicates of the supernatant liquid to Pyrex-glass centrifuge tubes and cautiously evaporate the bulk of the acetone. Evaporate similarly 1-ml duplicates of standard phenylbutazone soln. (3·0 mg per cent. in acetone). To each tube add 1 ml of *N* H_2SO_4 and heat in boiling water for 4 hr., restoring the vol. to 1 ml with water every 30 min. Chill the tubes in ice - water, add 0·5 ml of 0·5 per cent. $NaNO_2$ soln. and 2 ml of 1·8 per cent. *m*-toluidine ethyl sulphate (both reagents ice-cold) and mix after each addition. After 30 min. at room temp., add 0·2 ml of light petroleum (boiling range 40° to 60° C), shake briefly and centrifuge. Remove 3 ml of the aq. phase and determine the absorption at 525 mμ. H. F. W. KIRKPATRICK

1880. **Colloidal glass suspensions for use as standards for measurement of thymol turbidity [of blood serum].** J. G. Reinhold (*Anal. Chem.*, 1955, **27** [2], 239–244).—Factors affecting the prep. of a

colloidal glass suspension and its use in the calibration of photo-electric spectrophotometers for the thymol turbidity test have been studied. A suspension, suitable as a semi-permanent standard, is prepared by mechanical agitation for 8 to 12 hr. of clean borosilicate-glass fragments with distilled H_2O in a one-litre glass bottle. The milky liquid ($\simeq$ 200 ml) is decanted into a cylinder and made up to 1 litre with aq. 0·01 per cent. $HgCl_2$; the well-mixed suspension is put aside for 72 hr. and the upper 300 ml are then decanted into a stoppered and clean borosilicate-glass bottle. This suspension, which resembles more nearly than other standards the particle size and optical behaviour of the thymol - serum suspension, is standardised by further dilution with freshly distilled H_2O until the absorbance (measured at 660 mμ) is $\simeq$ 0·15. The readings of the turbid suspension produced, after 30 min., by the reaction at $\simeq$ 25° C of thymol - barbitone reagent (6 ml) with blood serum (0·1 ml) vary in spectrophotometers and photocolorimeters of different design. These variations are ascribed partly to the composition of the serum (flocculent or lactescent). When the glass suspension is used as a standard its turbidity (T_s) is given by A_s (T_m/A_m), where A_s = its absorbance at 660 mμ and T_m/A_m = mean ratio of thymol turbidity to absorbance for the particular spectrometer used. The thymol turbidity of a serum specimen is then equal to A (T_s/A_s), where A is the absorbance of the turbid suspension produced by the thymol - serum reaction. W. J. BAKER

1881. **Correction of paper-strip serum electrophoresis diagrams.** A. L. Latner (*J. Lab. Clin. Med.*, 1955, **45** [1], 147–148).—A description is given of the use of a Perspex-glass triangle for correcting the graph obtained when serum-protein concentration is plotted against the logarithm of the apparent optical density of stained electrophoresis patterns, when the graph, or the upper portion of it, is curvilinear. The device can be used for other investigations requiring the conversion of a curve to a straight line. W. H. C. SHAW

1882. **Paper chromatography of the non-volatile organic acids of biological fluids. II. The qualitative chromatogram from normal human urine.** R. Nordmann, O. Gauchery, J.-P. du Ruisseau, Y. Thomas and J. Nordmann (*Bull. Soc. Chim. Biol.*, 1954, **36** [11–12], 1641–1654).—Urine from healthy subjects is passed through a column of anion-exchange resin, chromatographed on paper in two directions with an acid and an alkaline solvent, respectively, and the spots are developed with bromocresol green. The R_F values of the spots are compared with those of 102 organic acids, which have been determined in the given solvents. Identification is checked by the addition of the suspected acid to the urine, followed by a further chromatogram. A variety of special developing reagents is listed that are specific for particular groups of organic acids. Normal urine gives 21 spots, of which one is due to oxalic, phosphoric and sulphuric acids, and others to fumaric, *m*-hydroxybenzoic, aconitic, glutaric, adipic, β-hydroxybutyric, hippuric, succinic, lactic, α-oxoglutaric, glycollic, malic, pyrrolidonecarboxylic, citric, tartaric, glucuronic, hydrochloric and uric acids; the remaining two spots arise from an unidentified acid and its hydrolysis product. Not all the spots are found in all samples of normal urine. H. P. PAGET

1883. **Problems of urine examination. Interference of sulphonamides in the detection of urobilinogen. Chemical detection of *B. coli*.** K. Borgmann (*Dtsch. ApothZtg.*, 1955, **95** [11], 263–264).—Sulphonamides interfere with the dimethylaminobenzaldehyde test for urobilinogen (Ehrlich test) in urine and must first be separated. *Procedure*—Urine (5 to 10 ml) is acidified with HCl and shaken with an equal volume of light petroleum. The petroleum layer is poured off, shaken with dilute alkali, and the aqueous phase tested with Ehrlich's reagent for urobilinogen. Reliable results with the Griess method for chemically detecting *B. coli* in urine are obtained only if the test is carried out in the following way. Mix equal parts of a 0·33 per cent. w/w soln. of sulphanilic acid in 30 per cent. acetic acid with a solution obtained by dissolving 0·2 g of 1-naphthoic acid in 20 g of water and diluting to a total weight of 150 g with 30 per cent. acetic acid. To the mixture add urine dropwise. If the urine contains *B. coli*, which reduces nitrate to nitrite, a raspberry-red colour is obtained owing to the formation of an azo dye. The colour is stable and is produced even if the urine is not fresh. P. S. STROSS

1884. **Determination of vitamin B_{12} in human serum by a mutant of *Escherichia coli*.** N. Grossowicz, J. Aronovitch and M. Rachmilewitz (*Proc. Soc. Exp. Biol. Med.*, 1954, **87** [3], 513–514).—A methionine-free casein hydrolysate medium was used which contained KH_2PO_4 (3·0 g), K_2HPO_4 (7·0 g), Na citrate trihydrate (0·5 g), $MgSO_4.7H_2O$ (0·1 g), $(NH_4)_2SO_4$ (0·05 g), dextrose (10 g), a 5 per cent. w/v soln. of methionine-free Casamino acids (100 ml) and water to 500 ml. The methionine-free soln. was prepared by taking a 10 per cent. soln. of Casamino acids (Difco) in *N* HCl and treating it with 0·05 *M* H_2O_2 for 24 hr. at room temp. Dextrose was sterilised separately as a 50 per cent. soln. and added aseptically to the cool sterile medium. Samples of serum were diluted, 1 to 10, with distilled water containing a trace of NaCN (0·5 μg per ml of serum) and heated to 100° C for 30 min. to free bound vitamin B_{12}. The medium was distributed in 2·5-ml amounts in test-tubes (18 mm × 150 mm) and autoclaved at 15 lb for 10 to 15 min. When the medium was cool, appropriate dilutions of heated serum or graded amounts of standard soln. were added and the vol. was made up to 5 ml with sterile water. The bacterial suspension was prepared by removing by loop some 24-hr. agar-slope culture, emulsifying in saline to a barely visible turbidity and inoculating each tube with one drop. After 40 to 48 hr. at 30° or 37° C, the growth was measured turbidimetrically at 620 mμ or 450 mμ. Normal subjects gave 200 to 1000 $\mu\mu$g per ml, those with pernicious anaemia gave 50 to 130 $\mu\mu$g per ml. H. F. W. KIRKPATRICK

1885. **Separation of cholesteryl esters in lipid extracts of animal tissues.** G. Clément, J. Clément and A. Louedec (*Arch. Sci. Physiol.*, 1954, **8**, 233–250).—Attempts were made to separate cholesteryl esters and glycerides by solvent pptn. of the esters, by selective hydrolysis of the glycerides with castor-oil-bean lipase and pancreatic lipase, and by chromatography on alumina columns. Quantitative separation was achieved only by the chromatographic technique, with light petroleum or CCl_4 as the eluting solvent on alumina activated by heating at 180° C. Artificial mixtures and the lipids of rat liver, rabbit liver, pig and human sera were effectively resolved into their glyceride and cholesteryl ester components by these methods.

NUTR. ABSTR. AND REV.

1886. **Quantitative cytochemical methods for the study of cell metabolism.** T. Caspersson (*Experientia*, 1955, **11** [2], 45–60).—The basis of ultra-microspectrography is described together with its application in the study of cell metabolism. The necessary instruments and equipment are detailed.
R. S. TONKS

1887. **Resolution of mixtures of amino acids by circular-paper chromatography.** N. A. N. Rao and T. K. Wadhwani (*Curr. Sci.*, 1954, **23** [11], 359–361).—An earlier method for the analysis of a mixture of amino acids by paper chromatography (*J. Ind. Inst. Sci.*, 1953, **35** [4], 343) was found to be time-consuming and not suitable for routine work. A mixture of amino acids can be resolved in 16 to 18 hours by circular-paper chromatography, in three different chromatograms, with the use of (*i*) *n*-butanol saturated with water at 20° C, (*ii*) a mixture of *n*-butanol, water and acetic acid (40:14:15), and (*iii*) a mixture of *n*-butanol, water and acetic acid (40 : 5 : 15).
R. J. MAGEE

1888. **Separation of amino acids on columns of sulphonated polystyrene resins.** P. N. Campbell, S. Jacobs and T. S. Work (*Chem. & Ind.*, 1955, [5], 117–118).—Zeo-Karb 225 ion-exchange resins are suitable for quant. estimation of the amino-acid composition of protein hydrolysates. The water-regain value of the resin is critical, and should lie between 1·55 and 2·3. A mixture of 15 amino acids (each 2×10^{-6} *M*) was separated on a 0·9-cm × 150-cm column of 200 to 400-mesh resin at 40° C, eluting with a soln. of sodium citrate of continuously varying pH (3·15 to 5·0) and concn. (0·2 *M* to 2 *M*) in 0·5 per cent. thiodiglycol and 0·05 per cent. BRIJ 35 detergent.
A. R. ROGERS

1889. **Amperometric determination of disulphides in intact proteins.** J. R. Carter (*Science*, 1954, **120** [3126], 895–896).—The amperometric method for −SH determinations (*Amer. J. Physiol.*, 1953, **173**, 109) can be adapted with modifications to the measurement of −S−S− in proteins by the reaction $RSSR + SO_3'' \rightleftharpoons RS' + RSSO_3'$. One ml of aq. soln. of protein treated with a minute amount of antifoam emulsion (Dow Corning AF) is added to 28 ml of 90 per cent. ethanol containing sufficient aq. NH_3, NH_4NO_3 and ethylenediaminetetra-acetate to make the concn. 0·25, 0·05 and 3×10^{-5} *M*, respectively, in a final vol. of 31 ml of reaction mixture; 2 ml of cold saturated Na_2SO_3 are then stirred in and titration with 10^{-3} *M* $AgNO_3$ is started immediately. It is essential that reagents are added in the order given, that the Na_2SO_3 ppt. remains in the titration mixture and that the Na_2SO_3 is freshly prepared and used within 45 min.
H. F. W. KIRKPATRICK

1890. **Ionophoresis of the adenosine phosphates.** D. C. Burke and A. B. Foster (*Chem. & Ind.*, 1955, [4], 94).—The three isomeric adenylic acids may be separated by ionophoresis on filter-paper in borate buffer at pH 10. Since there is no separation in a non-borate (acetate) buffer, the effect is attributed to different interactions of the isomers with borate ions. $C_{(2)}$:$C_{(3)}$-hydroxyl groups in the D-ribose portion of the 5′-phosphate interact strongly, giving a high migration rate; $C_{(3)}$:$C_{(5)}$-hydroxyl groups in the 2′-phosphate may interact weakly. No interconversion of isomers during ionophoresis was observed.
A. R. ROGERS

1891. **Critical evaluation of Feulgen microspectrophotometry for estimating amounts of deoxyribonucleic acid (DNA) in cell nuclei.** C. Leuchtenberger (*Science*, 1954, **120** [3129], 1022–1023).—The validity of the Feulger microspectrophotometric estimation of deoxyribonucleic acid (DNA) in cell nuclei has been tested by comparison of the results with those given by biochemical analysis, chromosome counts and u.v. spectrophotometry. The method can be utilised as a reliable means of comparison of relative amounts of DNA in nuclei of different cells, provided that the differences to be detected are sufficiently large.
H. F. W. KIRKPATRICK

1892. **The incorporation of radioactive acetate into biliary cholesterol and cholic acid.** [**Determination of bile acids.**] E. Staple and S. Gurin (*Biochim. Biophys. Acta*, 1954, **15** [3], 372–376).—A method is described for the isolation of bile acids from small quantities of bile containing 20 to 30 mg of bile acids. The method is based on that of Irvin, Johnston and Kopala (*J. Biol. Chem.*, 1944, **153**, 439). Proteins were pptd. from the daily bile sample by adding 3 volumes of 95 per cent. ethanol and, after being set aside at 5° C for 24 hr., the ppt. was washed with 95 per cent. ethanol. The washings and supernatant fluid were treated with NaOH and H_2O to give a final mixture containing 15 per cent. of NaOH and 50 per cent. of H_2O. After heating at 100° C for 24 hr., the ethanol was evaporated *in vacuo*, the residue was diluted with H_2O and extracted with ether; the extract was then washed, dried and evaporated. The residue was dissolved in ethanol and the cholesterol digitonide prepared. The aqueous phase from the ether extract was brought to pH 1·0 with aq. HCl and extracted with ether. The bile acids were extracted from this with 6·8 *N* HCl and, after adjusting the pH to 1·0, the bile acids were again extracted with ether. The residue was dried over P_2O_5 and identified by comparison with cholic acid and by chromatography (Bergström and Sjövall, *Brit. Abstr. C*, 1952, 497).
G. W. CAMBRIDGE

1893. **Use of sodium borohydride for determination of 3-oxobisnor-4-cholene-22-al.** E. H. Jensen and W. A. Struck (*Anal. Chem.*, 1955, **27** [2], 271–274).—A method for the determination of 3-oxobisnor-4-cholene-22-al, by reduction of the aldehyde group in alkaline medium with sodium borohydride, is described. The keto group also reacts, but at a much lower rate, and the aldehyde content can therefore be derived by zero extrapolation of a time-reduction curve. Excess of sodium borohydride is determined at intervals by treating an aliquot with KIO_3 and titrating the liberated iodine with $Na_2S_2O_3$. A standard deviation of ± 0·34 per cent. was obtained for samples in the 80 to 100 per cent. purity range.
G. P. COOK

1894. **Factors influencing the results of a starch-agar filter-paper disc method for determining alpha-amylase activity (and the effect of temperature on the nucleic acid content of growing mesophilic and thermophilic bacteria).** R. Wellerson, jun. (*Dissert. Abstr.*, 1954, **14**, 2182–2183).—The method consists in placing filter-paper discs on starch-agar plates, applying an exact amount of enzyme solution and incubating the plates at 52° C for six hours. The discs were removed, the plates were successively flooded with iodine solution and the zones read with a Fisher-Lilly zone reader. The optimum medium contained 1 per cent. of agar and 0·2 per cent. of sol. starch in *M*/15 Sorensen's buffer (pH 6·0). Storage of the medium at 12° C for a week did not improve the sensitivity of the method. Statistical analysis

of the zones of hydrolysis revealed that slight differences in the concentration of starch, concentration of agar, amount of enzyme per disc, pH of the medium, temp. of incubation and the length of the incubation period introduced significantly different zone diameters. Reproducibility of results was very good. The method is simple, accurate and allows the simultaneous testing of a large number of samples. Amylase activity was found to exhibit a definite linear relationship to the diameter of the zone of hydrolysis through a range of concentration of from 4·0 to 0·03 per cent. S.C.I. Abstr.

1895. **Estimation of plasma phosphatase by determination of hydrolysed phenol with amino-antipyrine [aminophenazone].** P. R. N. Kind and E. J. King (*J. Clin. Pathol.*, 1954, **7** [4], 322–326).—*Procedure*—Add 1 ml of buffer (pH 10 for alkaline, pH 4·9 for acid phosphatase) to 1 ml of substrate (0·01 *M* phenyl disodium phosphate), warm at 37° C for 3 min., add 0·1 ml of plasma, mix and incubate at 37° C for 15 min. (alkaline) or 60 min. (acid). For alkaline phosphatase add 0·8 ml of 0·5 *N* NaOH and 1·2 ml of 0·5 *M* $NaHCO_3$, and for acid phosphatase add 1·0 ml of 0·5 *N* NaOH and 1·0 ml of 0·5 *M* $NaHCO_3$. Add 1 ml of 0·6 per cent. aq. aminophenazone, mix, add 1 ml of 2·4 per cent. $K_3Fe(CN)_6$ and mix. Measure the colours with a green filter. A control expt. is carried out by this method, but plasma is added after the NaOH. Standards: add 1 ml of standard phenol soln. (0·01 or 0·03 mg of phenol) to 1·1 ml of buffer and then add reagents as for test. 0·01 mg of standard ≡ 10 King - Armstrong units. The buffer (pH 10) comprises 6·36 g of anhyd. Na_2CO_3 and 3·36 g of $NaHCO_3$ and water to 1 litre. The buffer (pH 4·9) consists of 21·0 g of cryst. citric acid dissolved in water and 188 ml of *N* NaOH; the vol. is made up to 500 ml with water, the pH being checked. A stock phenol soln. is prepared by dissolving 1 g in 1 litre of 0·1 *N* HCl (1 ml ≡ 1 mg) and this is diluted with water 1 to 100 or 3 to 100 for the working standards. Formol-stable acid phosphatase is determined by adding 0·02 ml of neutral 40 per cent. formaldehyde soln. to the buffer - substrate mixture of the standard and control.

H. F. W. Kirkpatrick

1896. **Chromatography of prostatic phosphatase.** H. G. Boman (*Biochim. Biophys. Acta*, 1955, **16**, 245–253).—Phosphatase activity was determined with 0·02 *M* *p*-nitrophenyl disodium phosphate as substrate (Bessey, Lowry and Brock, *Brit. Abstr. C*, 1946, 273), 200 µl being incubated with 10 µl of the enzyme fraction for 30 min. at 37° C and, after cooling, adding 0·1 *N* NaOH (2 ml). The nitrophenol liberated was estimated directly spectrophotometrically at 400 mµ. Total protein was also determined by measuring extinction of the fractions at 280 mµ. Purification of the prostatic phosphatase was carried out on an ion-exchange column (Dowex 50) by pH gradient elution chromatography; full details of the apparatus and technique are given. G. W. Cambridge

1897. **Quantitative estimation of succinic dehydrogenase activity in a single microscopic tissue section.** V. Defendi and B. Pearson (*J. Histochem. Cytochem.*, 1955, **3** [1], 61–69).—Sections of tissue were cut in the Linderström - Lang cryostat at a thickness of 5 µ, mounted on glass slides and incubated at 37° C for five min. in a substrate consisting of 5 ml of phosphate buffer at pH 7·4, 5 ml of 0·1 *M* sodium succinate and 5 ml of a soln. of 2-*p*-iodophenyl-3-*p*-nitrophenyl-5-phenyltetrazolium chloride (**I**) (1 mg per ml). The amount of reduced **I** is proportional to the activity of the enzyme present and to the area of the tissue. The section is dried and the reduced **I** is extracted by immersion in ethyl acetate (3 to 5 ml). The amount of reduced **I** is calculated by comparing the absorption at 494 mµ with standards prepared by reducing **I** with ascorbic acid.

N. E.

1898. **The oxidation of reduced xanthine dehydrogenase in chicken liver. [Determination of uric acid.]** D. B. Morell (*Biochim. Biophys. Acta*, 1955, **16** [2], 258–263).—Uric acid, produced by a xanthine dehydrogenase system in the presence of methylene blue as a hydrogen acceptor, was estimated by a modification of the Folin method (*J. Biol. Chem.*, 1933, **101**, 114). The methylene blue did not interfere with the colorimetric method, since it was almost completely adsorbed during the tungstic acid deproteinisation. Colour due to reaction with other substances was negligible.

G. W. Cambridge

1899. **Determination of xanthine oxidase in insects with tetrazolium salts.** A. D. Anderson and R. L. Patton (*Science*, 1954, **120** [3127], 956).—A description is given of a method in which two tetrazolium salts are used as acceptors in place of the methylene blue in the standard technique. These form stable coloured compounds, which can be extracted quantitatively and are therefore to be preferred to methylene blue.

H. F. W. Kirkpatrick

See also Abstracts 1807, 2011, 2013.

Drugs

1900. **Bromimetric determination of important medicinal compounds.** H. P. Kaufmann and W. Arends (*Arch. Pharm., Berlin*, 1954, **287** [9–10], 590–601).—A modified bromimetric method was applied to anethole, citral, terpineol, etc., essential oils (*e.g.*, anise, fennel, lavender), sulphonamides (*e.g.*, sulphanilamide, sulphacetamide, Globucid), purine derivatives (caffeine, theophylline) and to *p*-aminobenzoic esters (*e.g.*, benzocaine and procaine). A bromine solution in methanol, saturated with NaBr, is used. The substance (0·1 g) is dissolved in methanol, excess of the bromine solution is added and, after keeping in the dark for a period which depends on the substance being tested, the excess of Br is determined iodimetrically. The method gives better results for sulphonamides than the one with aqueous bromine solution. S.C.I. Abstr.

1901. **The determination of morphine in opium: ammonia, calcium hydroxide and shaking-out procedures.** K. Vellmer (*Dtsch. ApothZtg.*, 1955 **95** [11], 258–262).—The estimation of morphine in opium is critically reviewed. Only the method of Eder and Waeckerlin (*Quart. J. Pharm. Pharmacol.*, 1937, **10**, 680, and *Pharm. Acta Helv.*, 1940, **15**, 227) is found to give the true morphine content. Other methods, including those of the B.P. and U.S.P., are criticised, since the morphine is incompletely extracted and may also contain other opium alkaloids. If these more rapid methods are used, titration of the morphine gives rise to fewer errors than weighing. A methoxyl determination is used to assess the purity of the extracted morphine.

P. S. Stross

1902. **The identification of morphine, codeine, papaverine, thebaine, narcotine and tincture of opium.** A. A. Abdel Rahman (*Arch. Pharm., Berlin*, 1955,

288 [2], 53–57).—A drop of the solution containing 10 μg or less of opium alkaloid is spotted on to filter-paper (Whatman No. 1). A series of reagents are applied to this spot according to a scheme which is described. The opium alkaloids can be distinguished by the colour changes. The method is superior to spot-plate and test-tube tests, being more sensitive and making it easier to distinguish shades of colour. Conventional alkaloidal reagents such as sulphuric acid, nitric acid, ferric chloride, potassium ferricyanide - ferric chloride, Erdmann's, Froehde's, Mandelin's and Marquis's reagent can be used. The identification of tincture of opium is described. P. S. Stross

1903. **Distribution of anabasine between benzene and saturated aqueous solutions of sodium sulphate.** Kh. R. Rakhimov, S. F. Kulikov and S. N. Nabikhodzhaev (*Dokl. Akad. Nauk Uzb. SSR*, 1953, [7], 19–21; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 33,922).—The solubility of anabasine in 1 *M* Na_2SO_4 soln. at 25° C is 1·2 per cent. by wt. and in 1·5 *M* Na_2SO_4 soln. it is 0·75 per cent. When 1·5 *M* Na_2SO_4 is added to alkaline soln. of anabasine sulphate, 97 per cent. of the anabasine separates; a single extraction with benzene separates 99·95 per cent. of the anabasine. E. Hayes

1904. **Photometric determination of the content of hypericin in Herba Hyperici.** F. Neuwald and U. Hagenström (*Arch. Pharm., Berlin*, 1955, **288** [1], 38–42).—After a discussion of former colorimetric and photometric methods of determination of hypericin a modified procedure is recommended. The preparation of the drug and the examination of its extract by the use of a Pulfrich photometer with spectral filter S59 is described. A graph shows the extinction curve of a 2·75 mg per cent. solution containing pure cryst. hypericin in a 3 per cent. v/v solution of pyridine in methanol. In this medium, the optimal maximum for hypericin measurements is at 591 mμ. The highest contents of active substance are found in the leaves and flowers of the plant. The stems contain only 0·01 per cent. of hypericin. S.C.I. Abstr.

1905. **Chemical evaluation of dihydrostreptomycin, alone and in mixtures with procaine penicillin.** H. Vogt (*Arch. Pharm., Berlin*, 1955, **288** [1], 20–26).—It is shown that 1 mol. of dihydrostreptomycin base requires 3 mol. of reinecke acid to form an insoluble salt. Since this salt is not entirely insoluble in water, direct titration of the washed ppt. produces unsatisfactory results. An argentimetric method of determining the excess of ammonium reineckate left in solution is recommended. When the amount of active substance is being calculated, the water content in the dihydrostreptomycin salt must be taken into account. The technique can also be applied to mixtures of dihydrostreptomycin with procaine penicillin, after extraction of the latter with chloroform. S.C.I. Abstr.

1906. **The paper-chromatographic analysis of oestrogenic pharmaceutical preparations.** F. Markwardt (*Arch. Pharm., Berlin*, 1955, **288** [2], 82–86).—The oestrogen is extracted from the powdered tablet or oily solution with methanol or acetic acid. Papers are prepared by soaking in a solution of 5 per cent. of a silicone (Aquatex 4040) in *cyclo*hexane, draining off the excess and drying at 110° C. Spots of 5 to 50 μg of the oestrogen in a volatile solvent are applied and the chromatogram is developed (descending) with the lower layer of the mixture obtained by mixing 15 ml of water, 85 ml of methanol, 30 ml of benzene and 70 ml of hexane as the moving phase, the upper layer being used to saturate the atmosphere in the chamber. The paper is sprayed with a 10 per cent. solution of $SbCl_5$ in chloroform and heated to 90° C to show the position of the spots. Oestrone benzoate, stilboestrol dibenzoate and dienoestrol dibenzoate remain at the origin, while oestrone, β-oestradiol, ethinyl-oestradiol, stilboestrol and dienoestrol move with or near the solvent front. The last five can be separated if they are first acetylated. Good separations of oestrone acetate, oestradiol diacetate, oestradiol dipropionate, oestradiol 3-benzoate, stilboestrol diacetate, stilboestrol dipropionate, stilboestrol dimethyl ether, dienoestrol diacetate, dienoestrol dipropionate and dienoestrol dimethyl ether are obtained. P. S. Stross

1907. **The paper-chromatographic analysis of mixtures of caffeine, aspirin and phenacetin.** A. Castiglioni and M. Vietti (*Z. anal. Chem.*, 1955, **144** [2], 112–113).—Caffeine, aspirin and phenacetin (20 μg of each) may be separated by chromatography on Whatman No. 1 filter-paper in a mixture of butanol and aq. NH_3, *d* 0·880 (4 + 1). The R_F values by descending chromatography are: aspirin, 0·49; caffeine, 0·68; phenacetin, 0·93; and by ascending chromatography: aspirin, 0·48; caffeine, 0·67; phenacetin, 0·9. Aspirin is detected on the chromatogram by its fluorescence in u.v. light. Phenacetin gives an intense brown colour on exposure to iodine vapour. Caffeine may be detected by the yellow colour given with iodine vapour, or preferably by the intense yellow colour given with a 1 per cent. soln. of molybdophosphoric acid in dil. acetic acid. A. R. Rogers

1908. **Acidimetric determination of barbituric acid derivatives.** W. Poethke and D. Horn (*Arch. Pharm., Berlin*, 1954, **287** [9–10], 487–495).—A weighed amount (≃ 1 millimole) of barbituric acid derivative is dissolved in 24 ml of ethanol, and 0·1 per cent. alcoholic thymolphthalein solution and 15 ml of CO_2-free water are added. The solution is titrated with CO_2-free 0·1 *N* NaOH to the colour of an ammoniacal control solution containing 2 mg of Cu and 0·1 mg of Cr in 50 ml. The preparation of suitable control solutions, the selection of solvents and the determination of indicator error are discussed. The method was applied to phenobarbitone, barbitone, 5-(2-bromoallyl)-5-*iso*propylbarbituric acid (Noctal), methylphenobarbitone, etc., with an error of ± 0·6 per cent. S.C.I. Abstr.

1909. **Proposals for monographs for a number of barbituric acid derivatives which might appear in an addendum to D.A.B. 6 or in D.A.B. 7.** K. Winterfeld, K. Bodendorf and P. Loth (*Dtsch. ApothZtg.*, 1955, **95** [14], 324–327).—Detailed specifications of definition, description, solubility, tests of identity, tests of purity, assay and storage are suggested for 5-(2-bromoallyl)-5-*iso*propylbarbituric acid, 3-ethyl-5-*cyclo*hex-1′-enylbarbituric acid and its calcium salt, methylphenobarbitone, hexobarbitone and its sodium salt, and phenytoin. A. R. Rogers

1910. **Quantitative determination of thiobarbituric acids.** H. Wojahn and E. Wempe (*Arch. Pharm., Berlin*, 1955, **288** [1], 1–6).—Titrimetric determinations of 5:5-dialkylthiobarbituric acids (*e.g.*, thiopentone) are much quicker than the established gravimetric procedure and they are sufficiently accurate. The hypoiodite method, used for NH·C:S— compounds, can also be applied to thiobarbituric acids. It is based on the oxidation of

S to SO_4'' and a consumption of 8 equivalents of iodine per molecule. *Procedure*—To a freshly prepared solution of 50 mg of the sample in 100 ml of water and 5 ml of 20 per cent. NaOH are added 25 ml of 0·1 *N* iodine. After 10 to 15 min. the clear solution is acidified with 25 per cent. HCl and titrated to the point of decolorisation. Bromimetric and argentimetric methods are also applicable. S.C.I. ABSTR.

1911. **Some volumetric methods of determination of isoniazid.** E. Kühni, M. Jacob and H. Grossglauser (*Pharm. Acta Helv.*, 1954, **29** [8], 233–250).—Titration of isoniazid with 0·5 *N* $KBrO_3$ gives results $\simeq$ 0·5 per cent. too high. Titration with 0·05 *M* KIO_3 or 0·1 *N* I gives low results. The potentiometric titration of isoniazid in methyl cyanide with 0·1 *N* perchloric acid in dioxan gives two breaks corresponding to the dibasic compound. By the use of 0·1 *N* perchloric acid in acetic acid as titrant and acetic acid as solvent reproducible results are obtained only after acetylation of the isoniazid. The following procedure is recommended: Dissolve the sample (approx. 0·15 g previously dried at 103° to 105° C) by stirring in a mixture of acetic anhydride (2 ml) and acetic acid (18 ml); titrate potentiometrically with 0·1 *N* perchloric acid in acetic acid, or visually to an emerald-green end-point, with crystal violet as indicator.
A. R. ROGERS

1912. **Dicoumarol: some remarks on the quantitative determination.** K. Backe-Hansen (*Medd. Norsk. Farm. Selsk.*, 1954, **16** [7–8], 79–84).—Dicoumarol can be easily titrated as a dibasic acid in anhydrous pyridine with 0·1 to 0·2 *N* sodium methoxide in benzene - methanol (1 + 8) with thymol blue as indicator in an atmosphere of nitrogen; 1 ml of 0·1 *N* sodium methoxide $\equiv$ 0·01681 g of dicoumarol. N. E.

1913. **The polarography of some sulphur compounds. IV. The anodic waves of 2:3-dimercaptopropanol.** P. Zuman, R. Zumanová and J. Teisinger (*Coll. Czech. Chem. Comm.*, 1955, **20** [1], 139–146).—Polarography of 2:3-dimercaptopropanol at the dropping-mercury electrode gives an anodic wave attributed to the reaction $R(SH)_2 + Hg = RS_2Hg + 2H^+ + 2e$. The value of $E_{\frac{1}{2}}$ becomes regularly more negative with increasing pH over the range 2 to 8. An adsorption wave is also shown, with $E_{\frac{1}{2}}$ more negative in acid soln. and more positive in alkaline soln. than $E_{\frac{1}{2}}$ for the main wave. The sum of the heights of the two waves is proportional to concn. when this $<$ 0·0004 *M*. [This is a translation into German of a paper originally published in *Chem. Listy*, 1954, **48**, 1499.]
A. R. ROGERS

1914. **Furfural content of cigarette smoke and its determination.** E. W. Rice (*Proc. Soc. Exp. Biol. Med.*, 1954, **87** [3], 533–535).—With the standard technique of smoking and smoke collection (*Ind. Eng. Chem.*, 1936, **28**, 836), the furfuraldehyde is absorbed in xylene. The xylene soln. is extracted with aq. HCl (13 per cent. w/v), separated and centrifuged, dried with anhyd. sodium acetate, centrifuged and distilled. The furfuraldehyde is determined by the method of Rice (*Brit. Abstr. C*, 1952, 147). The results of determinations with various types of cigarettes are presented and discussed. H. F. W. KIRKPATRICK

1915. **The use of capillary analysis in the evaluation of galenical preparations.** A. Filip (*Československ. Farmac.*, 1953, **2** [9], 323–328; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 32,931).—The possible uses of capillary analysis for the evaluation of galenical preparations are reviewed and discussed.
E. HAYES

1916. **Measurement of entrained air in creams.** G. L. Blagg (*Pharm. J.*, 1955, **174**, 58).—An apparatus is described and illustrated for the determination of entrained air in pharmaceutical creams, emulsion paint, etc. The product is warmed to 40° C for about 15 min. in ethanol (95 per cent.), until it dissolves or is liquified, and the entrained air which is evolved is measured in a capillary tube.
S.C.I. ABSTR.

See also Abstracts 1817, 1822, 1823, 1947.

Food

1917. **Shortened procedure for routine assay of phosphorus in foodstuffs.** H. S. R. Desikachar (*J. Sci. Ind. Res. India, C*, 1955, **14** [1], 32–33).—A shortened procedure for the assay of P, which entails digestion with H_2SO_4 for 5 min. and colorimetric determination, measured about 95 per cent. of the total (determined by complete oxidation). Partially digested sugar or protein products do not interfere. R. J. COLE

1918. **Indirect determination of moisture in sugar products.** J. Terrier (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [6], 496–498).—The precision of the results of the determination of moisture in sugar products by the method described in *Brit. Abstr. C*, 1953, 512, is increased and the difference from the theoretical value is reduced to 0·2 per cent. by drying in a vacuum oven at 80° C and determining, by means of a blank, the correction to apply. R. J. MAGEE

1919. **Determination of metallic impurities in refined sugars.** M. R. Saunier (*Sucr. Franç.*, 1954, **95**, 399–402).—The dithizone method of determining lead in refined sugar is recommended in preference to the sodium sulphide method. The dithizone method showed an accuracy within $\pm$ 0·1 mg per kg for Pb concn. $<$ 0·5 mg per kg. The removal of organic impurities from low-grade sugars before determination of the Pb is shown to be advantageous. Determinations of Cu by the sodium diethyldithiocarbamate colorimetric method, of up to 20 mg of Zn per kg of sugar by a nephelometric method with the use of $K_4Fe(CN)_6$ in dilute acid, and of Sn by a colorimetric method with the use of dithiol in the presence of thioglycollic acid, are described.
SUGAR IND. ABSTR.

1920. **Colorimetric determination of small quantities of invert sugar in the presence of large excesses of sucrose.** J. Bernaerts (*Sucr. Belge*, 1954/1955, **74** [7–8], 165–171).—Nelson's reagent was prepared by dissolving 25 g of ammonium molybdate in 450 ml of water, adding 21 ml of conc. H_2SO_4, shaking and adding 3 g of $Na_2HAsO_4.7H_2O$ dissolved in 25 ml of water. The mixture was shaken and incubated at 37° C for 24 to 48 hours. The copper reagent was prepared by dissolving 50 g of cryst. sodium acetate in a 7 per cent. soln. of copper sulphate to make 100 ml. Sucrose (200 mg) in 2 ml of soln. was mixed with copper reagent (2 ml) in a 10-ml graduated tube and covered with 0·4 ml of *n*-butanol. The tube was stoppered and kept for 20 min. in a thermostat at 80° C. The mixture was cooled in cold water; Nelson's reagent (2 ml) and sulphuric acid (1·5 ml) were added and the volume made up to 10 ml with water. The

mixture was shaken continuously and an intense blue colour developed, which was measured in a Coleman nephocolorimeter with absorption cell 14–302A and filter 8–209 giving max. absorption at 525 mμ. The presence of certain salts, particularly phosphates, inhibits the colour formation, so the method is recommended only for sugars with a certain degree of purity. The conditions of pH and temp. in the reaction are arranged to prevent reduction of sucrose. Approximately 0·01 per cent. of invert sugar can be detected by this method.

SUGAR IND. ABSTR.

1921. **Quantitative invert-sugar determination with *m*-dinitrobenzene.** M. Kamoda (*Proc. Res. Soc. Japan Sugar Refineries' Technol.*, 1954, **3**, 145–150).—Sugar solution (5 ml) is mixed, with continuous shaking, with 0·5 per cent. *m*-dinitrobenzene solution in methanol (0·5 ml) and 1 per cent. NaOH solution (0·2 ml); the tube is placed in boiling water for exactly 4 min. and then cooled quickly to 0° C. The purple-red colour, which is linearly related to the invert-sugar concn., is measured in a spectrophotometer at 540 mμ. Optical densities were compared with those of standard invert-sugar solutions and gave results within a range of 0·001 to 0·01 per cent. The colour is relatively unstable. Sucrose does not react with the reagent nor does it inhibit its action, but galactose, mannose, arabinose, xylose and rhamnose interfere. Results by this method agree well with those by the Ofner method.

SUGAR IND. ABSTR.

1922. **Fermentation of sugars by an ultramicrotechnique prior to paper chromatography.** K. T. Williams and A. Bevenue (*Anal. Chem.*, 1955, **27** [2], 331).—A technique for the fermentation of small volumes of sugar soln. (0·01 to 0·05 ml) with commercial baker's yeast is described. The design of the micro test-tubes used allows direct application of the fermentation liquid to chromatographic paper.

G. P. COOK

1923. **The nature of the lead error in the polarisation of raw cane sugars.** L. Eynon, A. E. Tate and J. G. N. Gaskin (*Intern. Sugar J.*, 1955, **57** [1], 7–9).—A report on the lead error in the polarisation of raw sugars was presented at the International Commission for Uniform Methods of Sugar Analysis (Paris, 1954) (*Anal. Abstr.*, 1954, **1**, 2519). That report has been modified and replaced by the present report. Two conflicting theories of the nature of the error introduced during the clarification of raw cane sugar solution with wet and dry Pb acetate are discussed. In the case of wet lead defecation, experiment shows that, although the theories are in conflict, the practical results are not, but a similar comparison with dry lead defecation shows some considerable differences; it appears that the relationship between true polarisation and dry lead used is not valid for quantities of dry lead below 0·09 g. Gaskin has shown that the dry lead error can be calculated from the existing table by referring polarisation to minimum addition, *viz*, 0·09 g of dry lead. The Table III reported by Eynon (*Intern. Sugar J.*, 1954, p. 101) is now replaced; this new table has been adopted by the British National Committee for use in the U.K. from July 1st, 1955.

G. H. BAKER

1924. **Glucose and fructose contents of sugar-cane molasses.** F. H. C. Kelly (*J. Appl. Chem.*, 1954, **4** [11], 622–625).—The aldose groups were oxidised to aldonic acids by the method of Kline and Acree (*Ind. Eng. Chem., Anal. Ed.*, 1930, **2**, 413) except that 0·05 *N* iodine was used instead of 0·1 *N*. The technique involves alternate addition of 0·05 *N* iodine and 0·1 *N* NaOH, thus limiting the excess of either at any one time. The recovery of glucose from hydrolysed sucrose could fall as low as 90 per cent. for any one of the following reasons: too great an initial volume of solution, too short a time for completion of the reaction, or even the use of too much KI in the 0·1 *N* iodine (1 per cent. was sufficient). The residual fructose was determined by Luff's modified method (C. A. Browne and F. W. Zerban, "Physical and Chemical Methods of Sugar Analysis," 3rd Edn., John Wiley & Sons, Inc., New York, 1948, p. 382).

J. M. JACOBS

1925. **Filtration of crude fibre on filter-paper. Simplification of L. Bellucci's method.** H. Eschmann and M. Potterat (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [6], 499–501).—In the determination of crude fibre in foodstuffs, according to Bellucci (*Ann. Chim. Appl.*, 1932, **22**, 25), the replacement of the Gooch crucible by analytical filter-paper reduces the time of filtration to about one quarter.

R. J. MAGEE

1926. **Determination of the ageing of fish by means of iodimetric titration. II.** H. Truttwin (*Z. Lebensmitt Untersuch.*, 1954, **99** [6], 461–464).—A method for estimating the limiting value of palatability of plaice, codling, etc., held at, *e.g.*, temp. of 4° to 5° C, is discussed. Data are given on samples ranging from those that have a normal smell to those that have the smell of bad fish. A simplified time value test is given by using 1 g of fish, well teased out, and suspending it in 50 ml of water, to which are added 10 ml of 0·08 per cent. I solution, and the mixture is well shaken. For palatability in all fishes, the iodine is decolorised in 22 to 25 min.

S.C.I. ABSTR.

1927. **Detection of hydrogen peroxide in milk in presence of dichromate.** A. Roquette (*Ann. Falsif.*, 1955, **48** [553], 4–8).—By means of the perchromic acid reaction, H_2O_2 ($\leqslant$ 2 ml of 12-vol. soln. per litre) can be detected, provided that the sample (2 ml) is shaken with ether (2 ml) and dil H_2SO_4 (5 drops) immediately after addition of the dichromate. A blue tint is sometimes observable in the milk after addition of the dichromate, in which case the presence of H_2O_2 can be confirmed by the addition of the ether. The reaction is less sensitive to oxidising agents than that depending on the liberation of I from KI, but it gives negative results in presence of ClO_3', BrO_3', IO_3', S_2O_8'', Cr_2O_7'' and ClO'.

P. S. ARUP

1928. **Detection of nitrate in milk.** H. Hänni (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [6], 502–508).—The nitrate test with diphenylamine in sulphuric acid, as used in milk examination, is positive only for high nitrate concn. This difficulty is overcome by using the method of Hänni (*Mitt. Lebensmitt. Hyg., Bern*, 1951, **42**, 114), which detects as little as 2×10^{-5} per cent. of nitrate in milk. Milk strongly contaminated with cow-dung, or milk of animals with udder diseases or that of dairy cattle taking relatively high amounts of nitrates with fodder (beets, rape, etc.), showed no increase in nitrate content.

R. J. MAGEE

1929. **Detection of watering in boiled milk.** M. Staub and R. Krähenbühl (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [6], 477–483).—The influence of heating in open pans, refluxing and pasteurising on

the composition of milk is investigated. The margin of error for the various analytical methods used has been determined. By the determination of the fat-free dry substance, freezing-point lowering and refraction, the watering can be determined accurately only within 1 to 2 per cent. Tables of results for experiments with boiled, normal and watered milk are given. R. J. MAGEE

1930. **Biochemistry of cheese ripening. VIII. Proteinases of sour-milk cheeses and their activity in the ripening process. [Method of determination of proteinase activity in glycerin - water extracts.]** J. Schormüller, R. Wieske and H. Winter (*Z. LebensmittUntersuch.*, 1954, **99** [6], 438–460).—The micro-titration of the carboxyl groups according to the method of Grassmann and Heyde is adapted by using aq. 0·02 *N* to 0·05 *N* NaOH instead of alcoholic 0·01 *N* NaOH. Control of the end-point against a Cu solution and quick titration to eliminate the influence of CO_2 gives values of greater accuracy. The limit of error in the titration of 0·5 ml of a 6 per cent. solution of casein is ± 0·02 to 0·03 ml of 0·01 *N* KOH, which corresponds to 3 to 4 × 10^{-3} mg of amino-N. *Procedure*—The test solution, 0·5 ml, is mixed with 2 drops of a 0·5 per cent. thymolphthalein solution, and aq. 0·05 *N* NaOH is allowed to flow in until a clear blue colour is obtained (pH 9 to 10). To avoid pptn., 2 ml of absolute alcohol containing 0·02 per cent. of thymolphthalein are added and the blue colour disappears. From a 2-ml microburette graduated in 0·01 ml, 90 per cent. alcohol is added until the blue colour reappears. Absolute alcohol is added until the alcoholic concn. of the solution is 90 per cent., and the blue colour disappears. Finally the titration with aq. NaOH is made to a clear light-blue colour comparable with that of a 0·0025 *M* $CuCl_2$ soln. in excess of aq. NH_3. A blank obtained by titrating the same volume of 90 per cent. alcohol with 0·1 *N* KOH is subtracted; a white open box and the light of a 200-watt daylight lamp are used. S. C. I. ABSTR.

1931. **Ice-cream. Its composition, analysis and tentative standards.** S. N. Mitra and S. C. Roy (*J. Inst. Chem., India*, 1954, **26** [3], 159–170).—Local ice-creams have been analysed by the methods of A. L. Winton and K. B. Winton ("The Analysis of Foods," John Wiley & Sons, Inc., New York, 1945). The Kjeldahl determination can be replaced by determination of the albuminoid nitrogen (Mitra, *Brit. Abstr. C*, 1953, 481) with little loss of accuracy. G. B. THACKRAY

1932. **The chemical constituents of Victoria plums: preliminary qualitative analysis.** D. Dickinson and J. H. Gawler (*J. Sci. Food Agric.*, 1954, **5** [11], 525–529).—Methods are described for the preparation of extracts of Victoria plums and for the chromatographic examination of the extracts for anthocyanins and other colouring matters, chlorogenic and other acids, and sugars. An improved method of detecting certain acids after paper-chromatographic separation is described. After preliminary drying, the paper is sprayed lightly with iron solution [$FeCl_3$ (0·2 g) in 100 ml of ethanol (70 per cent.) acidified with a few drops of 0·1 *N* HCl] and then oversprayed with a 0·5 per cent. w/v solution of NH_4CNS in ethanol (70 per cent.). The acids (ascorbic, citric, malic, quinic, tartaric, lactic, aminoacetic, glutamic, oxalic and phosphoric) appear as white or yellowish spots on a speckled red background, the colour of which can be intensified by further spraying with the iron solution.
S. C. JOLLY

1933. **Estimation of tin in preserved foods.** A. R. Deschreider and L. van Coillie (*Lab. Central, Ministère Affaires Écon., Belgium*, Publ. 134, pp. 14).—The method of Cieleszky and Lindner (*Magyar Kém. Foly.*, 1951, **57**, 102) for the polarographic estimation of Sn was applied to a number of foodstuffs and found much more accurate, simple and rapid than previous analytical methods. Copper, iron and zinc in concn. of 10 to 20 μg per ml did not affect the estimation of Sn. Lead (1 to 2 p.p.m. or even up to 5 to 10 p.p.m.) did not affect the estimation when Sn was present in normal quantities. When Pb exceeded 5 p.p.m. and Sn was less than 50 p.p.m., two methods could be applied: either a standard curve from Pb in 12 per cent. HCl and a colorimetric estimation of Pb by dithizone enabled the concn. of Sn to be estimated from the curve of a mixture containing Sn and Pb, or the Sn could be completely eliminated from the curve by the action of ammonia and ammonium citrate.
NUTR. ABSTR. AND REV.

1934. **Variations of analytical results [of brewing materials].** M. Hengst (*Die Brauerei*, 1955, **9** [25–26], 154–156).—A statistical examination has been made of the variations in published results, mainly of the chief properties of malt. Variation may be due to inherent errors in the methods used, or to the frequency distribution of values for the population from which the samples are obtained. The probability of a value falling between certain limits can be assessed if error variation is kept to a minimum. Moisture results obtained by the vacuum-oven method are better in this respect than those obtained by the standard method. Determinations of the acrospire length are also unsatisfactory in this respect because pockets are formed in malt during turning; the samples are then obtained from several populations instead of one. Correlation between acrospire length and chemical results is poor.

Quarterly analytical results for 1948 to 1954 showed a lognormal distribution. By plotting them logarithmically against the frequency distribution calculated as a summed percentage of the total, curves approximating to straight lines are obtained where error variation is small. The median and 90 per cent. confidence limits for each constituent can be read off from the points of intersection of the curve by the 50 per cent., 5 per cent. and 95 per cent. ordinates, respectively. A factor can be calc. by which the median must be multiplied or divided to obtain the 5 per cent. and 95 per cent. limits, respectively. Values obtained by this procedure are, for malt-protein content, median 10·8 per cent., factor 1·09; soluble protein 35·6 per cent., 1·133; and extract 79·8 per cent., 1·019. These show good agreement with American results.
W. H. PARR

1935. **Determination of bitter substances in wort or beer.** P. Kolbach and H. Schilfarth (*Brauerei Wissensch. Beil.*, 1955, [1], 3–6).—Recent improvements in the chloroform-extraction method are described. A mixture of the wort or CO_2-free beer (150 ml), 96 per cent. ethanol (15 ml for wort, or 10 ml for beer), dil. (1 + 3) H_2SO_4 (1·5 ml), water (40 ml) and chloroform (30 ml, containing 1 per cent. of ethanol) is shaken during 1 hr. in a 350-ml stoppered flask (122 8-cm strokes per min.). After being set aside for 30 min. and then removing the aq. phase by suction, the chloroform layer is shaken for 15 min. with 100 ml of water, which is removed after setting aside for 15 min. The chloroform extract is now centrifuged (at 3500 r.p.m. for 15

min.) with 40 ml of water, and 15 ml of the clear extract are transferred to a small tared (microbalance) evaporating flask. An apparatus taking nine flasks comprises a wide glass tube in which the flasks stand on metal plates; the tube is externally steam-heated by means of a copper spiral tube. A current of dry CO_2 is sucked through the tube by means of a water-pump. During the evaporation of the chloroform, the pressure is maintained at $\leqslant$ 90 mm of Hg, and subsequently at 40 mm during 10 min. The residue is then weighed. Blank values are preferably determined on the original unhopped wort or beer, but approx. values may be taken at 27 mg per litre for 12 per cent. wort, and 25 mg for beer. A correction factor of 0·91 is applied to compensate for the chloroform (and its content of ethanol) which passes into the aq. phase during the extraction. Duplicate determinations show good agreement. P. S. ARUP

1936. **Determination of copper in hops and beer.** R. G. Ault, E. J. Hudson and A. G. R. Whitehouse (*J. Inst. Brewing*, 1955, **61** [1], 39–43).—For the following method, which is satisfactorily accurate and convenient for routine work, a $KMnO_4$ reagent is prepared by the addition of 70 g of $KMnO_4$ to 1·5 litres of boiling water, followed by 2 ml of aq. NH_3 (sp. gr. 0·880); the soln. is evaporated to 1 litre, cooled and the clear solution is decanted. Other reagents, *viz*, dil. H_2SO_4 (30 per cent. and 3 per cent., v/v) and aq. saturated $(NH_4)_2SO_3$ are freed from Cu by the usual treatment with Na diethyldithiocarbamate (**I**) and CCl_4. The beer sample (10 ml), mixed with 2 ml of 30 per cent. H_2SO_4, is boiled down to half bulk, cooled and then boiled with 5 ml of the $KMnO_4$ reagent; the treatment with the $KMnO_4$ reagent is repeated. After being cooled, a slight excess of aq. $(NH_4)_2SO_3$ is added. Hops (5 g) are stirred with 260 ml of boiling 3 per cent. H_2SO_4 (equiv. to 250 ml at room temp.), the mixture is cooled and occasionally stirred during 2 to 3 hr., after which 10 ml of the clear liquid are mixed with 2 ml of 30 per cent. H_2SO_4 and 5 ml of the $KMnO_4$ reagent, set aside for $\leqslant$ 1 hr., and then treated with a slight excess of aq. $(NH_4)_2SO_3$. The colorimetric determination of Cu in the soln. thus obtained from beer or hops proceeds as usual with the use of **I**, excepting that ethanolic 22·5 per cent. v/v CCl_4 (20 ml followed by 10 ml) is used, without shaking, for the extraction of the coloured copper compound. The combined extracts are washed with 2 ml of water. Attention is drawn to the uneven distribution of Cu in hops, and the consequent necessity for care in sampling.
P. S. ARUP

1937. **Comparative analyses of coffee extracts.** R. Casares and F. R. Alvarez Yguaran (*An. Bromatologia*, 1954, **6**, 365–373).—The caffeine contents, obtained by the method of Wyler and Högl, solids contents, ash contents, colour intensities, oxidation indices (defined as ml of 0·1 *N* $K_2Cr_2O_7$ needed to oxidise the steam-distillate from 1 g of coffee or the equivalent quantity of extract), chlorogenic acid and trigonelline contents of 12 commercial samples are presented. The standard methods used are described in detail. L. G. L. UNSTEAD-JOSS

1938. **Cacao polyphenolic substances. III. Separation and estimation on paper chromatograms.** W. G. C. Forsyth (*Biochem. J.*, 1955, **60** [1], 108–111).—Methods are described for the separation of the polyphenols of the cacao bean by two-dimensional paper chromatography, and for their estimation in extracts by paper chromatography followed by oxidation on the paper with 0·01 *N* $KMnO_4$.
J. N. ASHLEY

1939. **The quantitative determination of vanillin with the aid of 2:4-dinitrophenylhydrazine.** J. Meluzin (*Chem. Zvesti*, 1954, **8** [1], 22–25; *Referativnýĭ Zh., Khim.*, 1954, Abstr. No. 40,059).—The quant. pptn. of vanillin as the 2:4-dinitrophenylhydrazone is investigated. It is shown that the addition of NaCl before pptn. promotes the quant. pptn. of the hydrazone in a form that facilitates filtering and washing. E. HAYES

1940. **Some recent progress in the detection of adulteration of edible oils and fats.** S. N. Mitra (*J. Inst. Chem., India*, 1954, **26** [3], 138–145).—Methods of analysis of mustard oil, butter fat (ghee) and hydrogenated cooking fats (vanaspati) in use at the Bengal Public Health Laboratories are reviewed. G. B. THACKRAY

1941. **Identification of fats by urea fractionation.** W. F. Shipe (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 156–165).—Experimental procedure for the fractionation of fatty acids prepared from butter and other fats is detailed; three fractions, *A*, *B* and *C*, are obtained by the use of 10 per cent. or 20 per cent. urea solution, and the refractive indices of the samples of fatty acids so obtained are measured. Results obtained for various fats and oils are tabulated. Various differences provide evidence for the presence of individual fats; thus for butter fat the refractive indices of the *B* and *C* fractions are greater than that of the *A* fraction, whilst for coconut oil the *C* values are greater and the *B* values less than the *A* value. For the analysis of many samples, after eliminating grossly adulterated samples by determining the refractive index of the original oil, a screening technique is suggested.
A. A. ELDRIDGE

1942. **Saponification value determination of difficultly saponifiable drying-oil products.** J. N. Shaw and M. W. Formo (*J. Amer. Oil Chem. Soc.*, 1954, **31** [11], 448–451).—The use of *n*-butanol as a solvent for the saponification of a representative series of drying oils is described. By adding 5 per cent. of water to the *n*-butanol - KOH (40 g in 1 litre of solvent) reagent, results are obtained which compare well with calculated saponification values, whilst the rate of saponification in this solvent is much higher than in ethanol owing to the reflux temp. being higher by 40° C, and volatility losses are much less. The end-point in the *n*-butanol system is less sharp than in ethanol, but is improved if ethanol is added before titration. With the exception of maleic-modified oils of certain types, the solvent system of ethanol - pyridine shows excellent results based upon calculated values, but it has the disadvantage of being a two-solvent system, and has end-point difficulties. D. BAILEY

1943. **Paper-chromatographic separation of saturated fatty acids.** A. Holasek and K. Winsauer (*Monatsh. Chem.*, 1954, **85** [4], 796–801).—Saturated fatty acids, C_4 to C_{18}, can be separated by unidimensional ascending paper chromatography with the use of filter-paper steeped in 0·5 per cent. $KAl(SO_4)_2.12H_2O$ and dried at 60° to 100° C, and a mixture of CCl_4, methanol and conc. NH_3 soln. (81:18:1) for determining R_F values, after staining with Rhodamine B dissolved either in 0·05 *N* HCl or water and examining in u.v. light. The fatty acids are transferred on to the filter-paper in light

petroleum soln., which is allowed to evaporate before dipping the paper in the solvent mixture.
P. HAAS

1944. **Comparison of methods for determining fatty-acid oxidation produced by ultra-violet irradiation.** C. B. Kenaston, K. M. Wilbur, A. Ottolenghi and F. Bernheim (*J. Amer. Oil Chem. Soc.*, 1955, **32** [1], 33–35).—The thiobarbituric acid reaction for fatty-acid oxidation is compared with Lundberg and Chipault's method for peroxides, the Kreis test for aldehydes, and with the degree of conjugation, using fatty-acid esters exposed to u.v. light for various periods. The reaction is comparable with other methods for methyl linolenate and linoleate, but is relatively insensitive to the oxidation products of methyl oleate. The sensitivity of the thiobarbituric acid test for linolenate is 30 to 80 times that for linoleate at the same peroxide values (the corresponding values for other methods are 1·2 to 2·2).
D. BAILEY

1945. **Determination of the fatty-acid composition of lard triglycerides.** N. S. Drozdov and N. P. Materanskaya (*Tr. Mosk. Tekhnol. In-ta Myasnoĭ i Moloch. Prom-sti*, 1954, [2], 33–38; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 40,062).—Kaufmann's method of calculating the content of saturated acids, oleic and other olefinic acids, linoleic and other highly unsaturated acids in triglycerides is shown to be applicable to lard. It is as accurate as other methods of fat analysis.
E. HAYES

1946. **Rapid method for water-insoluble acids in butter.** L. G. Ensminger (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 183–184).—The sample of butter is shaken several times with hot water, then dissolved in ether and titrated with 0·05 *N* sodium ethoxide solution, phenolphthalein being used as indicator. Satisfactory results are recorded. The procedure is not suitable for the analysis of cream. The results compare well with those obtained by the official method ("Official Methods of Analysis," 7th Edn., A.O.A.C., Washington, D.C., 1950, p. 260) and is quicker.
A. A. ELDRIDGE

1947. **Sample preparation in the bioassay for vitamin D.** L. Friedman and G. M. Shue (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 165–172).—Stabilised vitamin D is incompletely extracted by the U.S.P. procedure of grinding with vegetable oil, and the sensitivity of the bioassay is diminished if, as in some infant foods, the mineral composition is abnormal and the vitamin-D content low. The evaporated milk or other sample is therefore heated under reflux with ethanolic KOH, cooled, diluted and extracted with ether; the extract is washed with water, then with NaCl solution, treated with anhydrous Na_2SO_4 and evaporated, the residue being dissolved in vegetable oil for feeding. Recoveries of vitamin D, when various extraction procedures are employed, are tabulated. Satisfactory results for various pharmaceutical products, foods, animal-feed supplements and milks are reported.
A. A. ELDRIDGE

1948. **The use of radioactive phosphorus in the assay of vitamin D.** P. Numerof, H. L. Sassaman, A. Rodgers and A. E. Schaefer (*J. Nutrition*, 1955, **55** [1], 13–21).—The procedure for vitamin-D assay suggested by Snyder, Eisner and Steenbock (*J. Nutrition*, 1951, **45**, 305), in which radioactive phosphorus is used, has been compared in concurrent assays with the U.S.P. line test carried out in two groups of rats, one of which received ^{32}P. The results showed satisfactory agreement. The subjective nature of the U.S.P. test is eliminated without any loss of accuracy.
N. E.

See also Abstracts 1884, 1985.

Sanitation

1949. **Paper chromatography for identifying air pollutants.** B. D. Tebbens and J. D. Torrey (*Science*, 1954, **120** [3121], 662–663).—An investigation for the presence of lower fatty acids in particulate form in air contamination was made with the use of the chromatographic method of Kennedy and Barker (*Brit. Abstr. C*, 1951, 410). Large vol. of air were drawn through a 15-cm Whatman No. 40 filter-paper on each of six days; the filters were extracted with dry ether in a Soxhlet apparatus or by steeping overnight at room temp., and the extracts were evaporated at room temp. to 15 ml, made up to 25 ml with ammonia-saturated ether and evaporated to small vol. for application to the paper. The samples were developed with 95 per cent. ethanol containing 1 ml per 100 ml of aq. NH_3 soln. by the ascending technique on Whatman No. 1 paper, air-dried for 1 hr. and then sprayed with bromophenol blue. A standard containing an equal wt. of the ammonium salts of formic, acetic, propionic and butyric acids was developed with each test. No sample contained an acid higher than acetic, but in several characteristic air-pollution episodes, acetate or formate was detectable by chromatography.
H. F. W. KIRKPATRICK

1950. **The volumetric determination of small amounts of bromides and iodides in water supplies and mineral waters.** P. Höfer (*Gesundheitsing.*, 1953, **74**, 224–226).—A procedure is given for the determination of bromides in drinking water or mineral waters, the method being based on the oxidation of Br' to BrO_3' by hypochlorite in the presence of Cl' at a pH value of 6·5 to 7·2, which is maintained by adding a mixture of mono-, di- and pyro-phosphates. Interference by Fe is reduced in the presence of pyrophosphate. The BrO_3' is then estimated iodimetrically. The method is applicable to concn. from 0·16 mg per litre to 15 mg per litre with an accuracy better than 10 per cent. A procedure is also given for the determination of iodides and bromides in the presence of one another, based on differences in the oxidation potential of iodate and bromate. The iodate is determined at a pH value of $\not< 3$ and the bromate by acidifying with formic acid to a pH value of 1. Nitrites do not interfere.
WATER POLLUTION ABSTR.

1951. **A micro-method for the determination of organic carbon in lake water.** I. Forsblad (*Mikrochim. Acta*, 1955, [1], 176–186).—An apparatus is described for the continuous evaporation of lake water *in vacuo*. The evaporation is carried out in the presence of H_3PO_4 to eliminate Cl' and CO_3''. The organic residue is then oxidised in the evaporating flask in 3 min. at 160° C with a mixture of KIO_3 (1·5 g), CrO_3 (15 g), P_2O_5 (30 g), conc. H_2SO_4 (130 ml) and 90 per cent. H_3PO_4 (25 ml). The CO_2 produced is absorbed in 0·025 *M* $Ba(OH)_2$ *in vacuo*, and the resulting carbonate is titrated between pH 10 and 3·5, with thymolphthalein and bromophenol blue as indicators. The accuracy is $\pm$ 1 per cent. with samples containing 2 mg of organic matter. Results on lake water are given.
K. J. GARDNER

1952. **Colorimetric determination of small quantities of methylenedioxyphenyl-containing pyrethrum synergists.** M. S. Blum (*J. Agric. Food Chem.*, 1955,

3 [2], 122–124).—In the presence of conc. gallic and sulphuric acids, methylenedioxyphenyl compounds produce a green complex, which is stable for several hours and obeys Beer's law. *Procedure*—To 10 mg of gallic acid in a dry tube is added by pipette 0·1 ml of methyl cyanide containing 5 to 80 μg of the methylenedioxyphenyl-containing synergist (**I**). By means of a burette, 3 ml of conc. sulphuric acid are run in to form a layer; after 1 min., the contents are mixed thoroughly. A blank is prepared with **I** omitted. After 2 hr. the optical density is determined at 660 mμ and the concentration of **I** is read from a previously prepared standard graph.

H. B. HEATH

1953. **Insecticide mixture analysis. Chromatographic separation of dichlorodiphenyltrichloroethane [DDT] and dichlorodiphenyldichloroethane [DDD] mixtures.** A. R. Amell and R. Helt (*J. Agric. Food Chem.*, 1955, **3** [1], 53–55).—A simple and accurate chromatographic method is described for the analysis of mixtures of DDT and DDD. *Procedure*—The sample, together with weighed amounts (not stated) of the two dyes 1-xylylazoxylylazo-2-naphthol (D & C Red No. 18) and 1-hydroxy-4-*p*-toluidinoanthraquinone (D & C Violet No. 2), is dissolved in *n*-hexane saturated with nitromethane and the solution is passed under pressure through a column (18 mm × 125 cm) containing 100 g of a gel of silicic acid, previously packed into the column by dispersing the acid in 300 ml of the solvent and forcing the solvent through the column under a pressure of 100 mm of nitrogen. The chromatogram is developed with a total of 500 ml of solvent and the eluted fractions containing the red dye (and the DDT) and violet dye (and the DDD) are collected separately. The solvent is removed from each fraction and the residue is cooled and weighed. The amounts of the insecticides in the mixture are obtained by subtracting the weight of dye used from the residues. Results are correct to within ± 1 per cent.

S. C. JOLLY

1954. **Simplified method of estimating DDT [dicophane] residues.** R. C. Amsden and D. J. Walbridge (*J. Agric. Food Chem.*, 1954, **2** [26], 1323–1324).—Wipe the surface on which the estimation is to be performed with a cotton swab washed with ether. Wash the cotton with ether, using as little solvent as possible; evaporate the solvent. Add 3 to 4 ml of a 1 + 1 mixture of fuming HNO_3 - conc. H_2SO_4. Immerse in boiling water, remove and cool 2·5 min. after brown fumes are evolved, and wash into a stoppered test-tube containing 10 ml of water. Cool, add 10 ml of benzene and shake vigorously for 0·5 min. Remove 1 ml of benzene with a pipette, add 3 ml of a mixture of benzene and *iso*propylamine (3 + 1). Shake gently and compare the yellow colour with $K_2Cr_2O_7$ standards prepared to match known quantities of dicophane.

L. G. L. UNSTEAD-JOSS

See also Abstracts 1769, 1791, 1805.

Agriculture and Plant Biochemistry

1955. **Test for pectin based on reaction of hydroxamic acids with ferric ion.** R. M. McCready and R. M. Reeve (*J. Agric. Food Chem.*, 1955, **3** [3], 260–262).—A test for pectin, apparently specific under the experimental conditions, which can be used as a histochemical test on plant tissues is described. *Procedure*—To the sample (about 5 mg) dissolved or suspended in 1 ml of water, 1·0 ml of 13·9 per cent. w/v aq. hydroxylamine solution is added, followed by 1 ml of 14·0 per cent. w/v aq. NaOH solution. After 2 min. at room temp., 1 ml of aq. HCl (1 vol. of conc. HCl diluted with 2 vol. of water) is added, followed by 1 ml of $FeCl_3$ reagent (10 g of the hexahydrate dissolved in 100 ml of 0·1 *N* HCl). A positive reaction is indicated by a red ppt. A control is carried out by adding the sample to the reagents after the addition of the HCl. For the application to plant tissues, sections (80 to 520 mμ thick and placed in water containing 300 p.p.m. of SO_2 immediately after cutting) are tested in 10-ml dishes with the use of double the above quantities of each reagent and, after colour development (1 min.), the excess of the reagents is washed out with water and the section examined at magnifications (≃ × 1200), after vacuum infiltration with water to remove the profuse gas bubbles formed during the reaction.

S. C. JOLLY

1956. **The influence of the method of determination of lignin on the lignin-ratio technique for digestibility in the cow.** D. A. Balch, C. C. Balch and S. J. Rowland (*J. Sci. Food Agric.*, 1954, **5** [12], 584–588).—Three methods for the determination of lignin (Armitage *et al.*, *J. Soc. Chem. Ind.*, 1948, **67**, 241; Ellis *et al.*, *J. Anim. Sci.*, 1946, **5**, 285; Norman and Jenkins, *Biochem. J.*, 1934, **28**, 2147, 2160) have been compared on samples of food and faeces. The method of Armitage *et al.* gave the lowest values for crude lignin (*L*), nitrogen content of *L*, and lignin corrected for nitrogen content. The mean digestibility coefficients (*C*) for *L* and corrected lignin determined by the three methods were: 1·3 ± 0·8 and 4·4 ± 1·0, 4·7 ± 0·6 and 9·1 ± 0·7, and 9·3 ± 0·7 and 8·3 ± 0·5, respectively. *L*, as determined by the method of Armitage *et al.*, should be acceptable as the basis of a ratio technique. In a series of digestibility trials with cows on hay - concentrate diets, *C* for *L* was − 0·72 ± 0·7.

S. C. JOLLY

1957. **The quantitative determination of guttapercha in plants.** M. Rychnovská-Soudková (*Československ. Biol.*, 1953, **2** [5], 305–307; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 32,933).—A sample (140 to 255 mg) of dried ground plant material is extracted with benzene, the extract is evaporated to dryness and the residue is treated with 25 ml of 96 per cent. ethanol and set aside for 24 hr. The film of guttapercha is filtered off, dried at 40° C and then dried in a desiccator until the wt. is constant. The precision is ± 1 per cent.

E. HAYES

1958. **Colorimetric method for the determination of molybdenum in soils.** C. H. Williams (*J. Sci. Food Agric.*, 1955, **6** [2], 104–110).—A rapid and accurate method is described for determination of Mo in soil. None of the elements commonly found in soil at normal concn. is likely to interfere. *Procedure*—An aliquot of the soil extract (equiv. to ≃ 1 g of soil) is evaporated to dryness and, if necessary, ignited at a temp. ⩾ 450° C to destroy organic matter. The residue is dissolved in 5 ml of conc. HCl, and the solution is diluted to 20 ml in a stoppered test-tube (8 in. × 1 in.). After shaking for 1 min. with 10 ml of purified dithizone solution [5 g dissolved in 500 ml of $CHCl_3$ is extracted with 1 litre of dilute aq. NH_3 (10 ml of conc. aq. NH_3 per litre) and the aq. extract is washed with three 100-ml portions of redistilled $CHCl_3$; after slight acidification of the aq. solution with redistilled HCl, the dithizone is extracted with 1 litre of redistilled $CHCl_3$ and the extract is washed with three 200-ml portions of

water; the solution is stored at temp. < 4° C], the lower $CHCl_3$ layer is siphoned off. The tube is washed down with a minimum of water, and 5 ml of conc. HCl, 3 ml of 5 per cent. KCNS solution and 5 ml of 4 per cent. ascorbic acid solution are added, mixing between each addition. After the red colour of $Fe(CNS)_3$ has faded completely (10 to 15 min.), 15 ml of ether are added and, after shaking for 1 min., the lower aq. layer is siphoned off and the tube washed down with the ether. The ether is evaporated off from the solution and the residue is digested with 3 ml of conc. H_2SO_4, 1 ml of $FeSO_4$ solution (0·5 mg of Fe per ml) and 6 drops of perchloric acid (70 per cent.) until the digest is quite clear. The cold solution is diluted with 15 ml of water and heated in boiling water for 10 to 15 min. to dissolve any $Fe_2(SO_4)_3$. The Mo in the solution is determined by pptn. and extraction of the dithiol complex as described by Piper and Beckwith (*Brit. Abstr. C*, 1949, 104). For soils high in Fe, a sample containing ⩾ 150 mg of Fe must be used.

S. C. JOLLY

1959. **Potash determination in fertilisers. Flame-photometric determination with ion-exchange separation of interfering anions.** C. W. Gehrke, H. E. Affsprung and E. L. Wood (*J. Agric. Food Chem.*, 1955, **3** [1], 48–50).—A rapid and accurate method is described for the determination of K in fertilisers. *Procedure*—The sample (1·5 g) is boiled for 5 min. with 100 ml of water and 15 ml of 1·5 *M* ammonium carbonate; 10 ml of aq. NH_3 soln. (1 + 1) are added, the mixture is allowed to cool, diluted to 100 ml and filtered. To a 20- or 25-ml aliquot (for > 30 or < 30 per cent. of K_2O in the sample, respectively), adjusted to pH ≃ 5 with HCl (methyl red indicator), sufficient (7 to 10 ml of wet resin) Amberlite IR–4B (Cl) (regenerated to the chloride form by exhausting with 5 per cent. Na_2CO_3 or NaOH soln., washing free from base, treating with 3 portions of HCl, and washing with water) is added. After being shaken continuously for 5 min. (or intermittently for 15 min.), the mixture is filtered through a high-speed filter-paper and the resin is washed at least 3 times with water. The combined filtrate and washings are diluted to 250 ml, 500 ml or to 1 litre (for ⩾ 13, ⩾ 40 or > 40 per cent. of K_2O, respectively, in the sample), and the amount of K present is determined by comparing the meter deflection given by the sample in a flame spectrophotometer, using a red-sensitive phototube, with that given by a standard solution of KCl whose K_2O content is approx. the same as that of the sample. This procedure effectively removes interfering anions and cations commonly present in commercial fertilisers. The average standard deviation is ± 0·15 per cent. and the method is as accurate as the A.O.A.C. gravimetric method.

S. C. JOLLY

1960. **Reliability of chemical analyses for fertilisers and feeds.** S. R. Miles and F. W. Quackenbush (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 108–130).—Factors affecting the precision of chemical analyses of commercial lots of fertiliser or feed are discussed, and sample analyses are tabulated. Deviations of results for N, P and K in fertilisers, and of protein, fat, fibre and ash in feeds, under various conditions, are also tabulated. A. A. ELDRIDGE

1961. **Note on the catalyst in the Kjeldahl procedure for nitrogen in fertilisers.** H. R. Allen (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 185).—For routine determinations of nitrogen in fertilisers, a catalyst composed of 5 parts of copper sulphate (dried at 110° C) and 1 part of selenium powder is placed in the digestion flask before digestion is started. K_2SO_4 is not used. Unsatisfactory samples are rechecked with the use of Hg or $CuSO_4$ with K_2SO_4, adding the catalyst after the preliminary digestion. In the analysis of organic materials, the modified procedure gives results ≃ 0·1 per cent. lower than those obtained by the usual procedure.

A. A. ELDRIDGE

1962. **Estimation of fused and sintered phosphatic fertilisers by X-ray diffraction.** E. Asada (*Japan Analyst*, 1953, **2** [1], 17–21).—Essential data for the X-ray diffraction study of fused and sintered phosphatic fertilisers are summarised, and a few of these products are examined by means of them. Both fused (1500° C) and sintered (1350° C) phosphatic fertilisers made from apatite and SiO_2 consist of α-$Ca_3P_2O_8$ and solid soln. of CaO and SiO_2 in that phase. The solubility of sintered magnesia phosphatic fertiliser in citric acid is not due to α-$Ca_3P_2O_8$ or other crystalline substance soluble in citric acid, but to a non-crystalline glass. The residue shows a diffraction pattern similar to that of β-$Ca_3P_2O_8$. Thus the addition of MgO appears not only to decrease the m.p., but also to retard crystallisation, so that the formation of glass which is less resistant to acid is promoted. K. SAITO

1963. **Chemical method for measuring relative digestibility of animal protein feedstuffs.** A. J. Gehrt, M. J. Caldwell and W. P. Elmslie (*J. Agric. Food Chem.*, 1955, **3** [2], 159–162).—A method of determining the amounts and nature of indigestible fractions of meat and fish by-products that are used as feeding-stuffs is described. The fat is first removed from the sample by ether-extraction, followed by separation of meat and light impurities from bone and heavy impurities by flotation with CCl_4. The meat fraction is digested with 0·1 *N* HCl containing 0·2 per cent. of pepsin at 37° C for 48 hr. The indigestible fraction is separated by centrifuging and consists of hair, vegetable fibre and colloidal organic matter. The heavy residue from the flotation is treated with HCl to remove bone; heavy impurities such as glass and sand are left.

H. B. HEATH

1964. **Determination of small amounts of diethylstilboestrol [stilboestrol] in feeds.** E. W. Cheng and W. Burroughs (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 146–150).—The sample (prepared by mixing a solution of stilboestrol in corn oil with soya-bean-oil meal) was extracted with benzene, the concentrated extract was mixed with Skellysolve B, and the mixture was then passed through a column of Celite wetted with NaOH and Skellysolve B. The stilboestrol, which is retained, was eluted with alcoholic HCl and determined either colorimetrically with a solution of $SbCl_5$ in ethylene chloride, or after evaporation and dissolution in acetic acid and irradiation with ultra-violet light, by determination of absorbance. A Coleman colorimeter, with filters, 525 and 430 mμ, respectively, was used. The $SbCl_5$ reaction method is the more sensitive; average recoveries by the two methods were, respectively, 98 and 94 per cent. Some interfering substances may not be removed by the chromatographic procedure, and the method is not necessarily applicable to other mixes without modification.

A. A. ELDRIDGE

1965. **The determination of moisture in gluten and sweetened feeds.** W. R. Fetzer and L. C. Kirst (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 130–140).—Procedures for the determination of moisture in a

component of a feeding-stuff may not be satisfactory when applied to the compounded material. For heat-sensitive mixtures or materials, *e.g.*, gluten feed or sweetened feed, distillation with benzene gives more accurate results than that with toluene. The sweetened feed, mixed with water and Filtercel, may also be heated in a vacuum-oven at 70° C for 20 hr. to give a reproducible loss in weight.

A. A. ELDRIDGE

1966. **Determination of Nicarbazin in feeds.** C. R. Szalkowski, M. G. O'Brien, C. W. Stewart and W. J. Mader (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 140–146).—Nicarbazin (a complex of 4:4′-dinitrocarbanilide with 2-hydroxy-4:6-dimethylpyrimidine) is extracted from chicken feed with hot dimethylformamide. Substances that resemble dinitrocarbanilide in giving a yellow colour with alcoholic NaOH, and inhibit the colour reaction of hydroxydimethylpyrimidine with sulphanilic and nitrous acids, are removed chromatographically with Al_2O_3. The procedure for the spectrophotometric determination of the two components is detailed. Recoveries of 97·0 to 103·0 per cent. of Nicarbazin added to blended feeding-stuffs are reported.

A. A. ELDRIDGE

1967. **Rapid test for the detection of Toxaphene in agricultural formulations.** D. P. Johnson (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 153–156).—The Toxaphene is extracted from the insecticide by shaking with *n*-hexane. A mixture of the extract with pyridine and methanolic *N* KOH is heated at 100° C for 15 sec., then cooled at 0° for 1 min. If Toxaphene is present, a pink to rust-red colour develops within 10 sec. after heat is applied. If S is present, the sample is first extracted at 0° with methanol, and the residue, after evaporation, is employed for the test. Interference by parathion can be prevented by washing the hexane extract with aq. Na_2CO_3. Some interfering materials may be eliminated by partition chromatography, with *n*-hexane and nitromethane as solvents and silicic acid as the supporting medium. A. A. ELDRIDGE

1968. **Herbicide determination. Determination of 2:4-dichlorophenoxyacetic acid (2:4-D) in grain and seed.** R. P. Marquardt and E. N. Luce (*J. Agric. Food Chem.*, 1955, **3** [1], 51–53).—A colorimetric method is described for the determination of as little as 25 μg of 2:4-D per 100 g of sample. *Procedure*—The sample (200 g as a fine meal) is shaken continuously for 1 hr. with a mixture of 500 ml each of chloroform and ether and 10 ml of acetic acid. The mixture is filtered and the residue washed with a mixture of 50 ml each of chloroform and ether. The combined filtrate and washings are extracted with NaOH soln. (once with 200 ml of water and 50 ml of 5 *N* NaOH and twice with 200 ml of water and 10 ml of 5 *N* NaOH) and the combined extracts are washed with three 100-ml portions of chloroform. The aq. solution is acidified with 50 ml of conc. HCl and extracted with 125- and 100-ml portions of ether. The combined filtered ethereal extracts are extracted with 50-, 25- and 25-ml portions of 0·5 *N* NaOH. The combined aq. extracts are washed with three 10-ml portions of chloroform, acidified with 10 ml of conc. HCl, and 5 ml of 40 per cent. w/v aq. solution of tungstophosphoric acid are added. The mixture is extracted with three 10-ml portions of chloroform, and the combined filtered extracts are percolated through a column (17 mm by 20 cm) of Hyflo Super-Cel. The column is then washed with 150 ml of chloroform containing 3 per cent. v/v of methanol, at an air pressure that gives a percolation rate of 150 ml per hr., and then with 25 ml of chloroform containing 7 per cent. v/v of methanol; the washings are discarded. The eluate from the percolation of a further 150 ml of chloroform containing 7 per cent. v/v of methanol is collected, and extracted with three 25-ml portions of extraction solution (19·0 g of $Na_2HPO_4.7H_2O$ and 10·0 g of $NaH_2PO_4.H_2O$ per litre; pH 6·85). The combined extracts are washed with three 10-ml portions of chloroform, then acidified with 2 ml of conc. HCl and extracted with three 10-ml portions of chloroform. The combined extracts are decanted from any separated water globules and evaporated just to dryness on a water bath. To the cooled residue, 50 ml of chromotropic acid reagent (0·10 g of the Na salt dissolved in 100 ml of conc. H_2SO_4) are added, and the mixture is heated at 150° ± 2° C for exactly 10 min. The cooled solution is poured into 30 ml of H_2SO_4–stannous chloride solution (**I**) (50 g of $SnCl_2.2H_2O$ dissolved in a cold mixture of 200 ml of conc. H_2SO_4 and 800 ml of water), the mixture is cooled and, after about 5 min., diluted to 50 ml with **I**. The mixture is filtered and, 30 to 45 min. after first adding to **I**, the transmittancy of the filtrate at 565 mμ is measured in a 5-cm cell against water. The amount of 2:4-D is derived by reference to a standard curve prepared from samples of untreated meal to which known amounts of 2:4-D have been added. S. C. JOLLY

1969. **Paper chromatography of some organic phosphate insecticides. IV. Spot test for *in vitro* cholinesterase inhibitors.** J. W. Cook (*J. Ass. Off. Agric. Chem.*, 1955, **38** [1], 150–153).—Visual location of the inhibitors along the chromatogram of organic phosphate pesticides (*Ibid.*, 1954, **37**, 989) is accomplished by placing in contact four prepared paper strips in the following order starting from the top: substrate, enzyme plus bromothymol blue, chromatogram, water. The enzyme preparation consists of alkaline blood plasma, and the substrate is acetylcholine chloride. Tests with organic phosphate insecticides show that the technique is frequently highly sensitive. A. A. ELDRIDGE

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

1970. **Detection of micro quantities of halogens by means of the Beilstein test.** H. Jurány (*Mikrochim. Acta*, 1955, [1], 134–139).—An apparatus is described that allows the Beilstein test for halogens to be used for very small quantities of organic compounds. It comprises a piece of glass tubing (8 mm × 10 cm) one end of which is connected to a gas supply, whilst the other end is drawn out to hold a copper tube (15 mm × 1 mm). The gas is ignited at the open end of the copper tube after the substance under test has been placed in the glass tube, which is then heated to volatilise the sample. If halogen is present, the flame at the end of the copper tube is green. K. J. GARDNER

1971. **A study on the autotitrator.** T. Takahashi, E. Niki, K. Kimoto and W. Yoshimura (*Japan Analyst*, 1953, **2** [2], 126–133).—An automatic glass-stoppered titrator is set up with the aid of a d.c.

amplifier and a relay, and its application to potentiometric titration is studied. For a neutralisation titration, an antimony electrode is used, which works well for neutralisations involving a weak acid or base. For the titration of iodine, a dead-stop method with platinum electrodes can be used, but not when standard iodine solution is the titrant.
K. SAITO

1972. **New technique for two-dimensional descending paper chromatography.** M. Yamaguchi and F. D. Howard (*Anal. Chem.*, 1955, **27** [2], 332–333).—To obviate the use of large cabinets with corrosion-resistant linings, a method of conducting paper chromatography inside a borosilicate-glass jar (24 in. × 12 in.) has been devised. The solvent tray is a borosilicate-glass dish (6 in. diam., with non-fluted edge) resting in a 5-in. funnel that is fitted into a 250-ml graduated cylinder so that the height can be adjusted to suit the length of paper. The cylinder rests on a glass plate, the glass jar being inverted over the entire assembly and the joint being made air-tight. The procedure for obtaining the chromatogram, and the preparation of the filter-paper sheet (22½ in. × 18½ in.) for first and second dimensions, are described in detail.
W. J. BAKER

1973. **Constant-volume [chromatographic] fraction collectors.** J. W. Hahn and M. Nyman (*Anal. Chem.*, 1955, **27** [2], 330–331).—A constant-vol. fraction collector (min. vol. $\simeq$ 1·5 ml) for use in column chromatography was constructed by adding a latch-relay and a siphon on a balance-arm to the motor-driven turntable of a time-actuated collector. The wt. of solvent and the movement of the balance-arm initiates the action, a single impulse indexing the table one tube only. A similar, but optically controlled, collector was made by rigidly fixing this type of siphon so that the delivery arm interrupts a light-beam impinging on a photocell. When the siphon delivers and solvent passes through the glass tube, more light is transmitted than when the tube is empty. This effect, suitably amplified, energises a solenoid - ratchet device that turns the table. The full circuit for each collector is shown, and precautions to be taken during their continuous operation are noted.
W. J. BAKER

1974. **Fraction-collection apparatus.** Technicon Chromatography Corporation (Brit. Pat. 725,736, Date Appl. 6.5.53).—An apparatus for automatically collecting chromatographic fractions is described and illustrated. The operating mechanism of the apparatus is described in detail.
D. BAILEY

1975. **Continuous detection and measurement of low concentrations of oxygen in gases.** L. P. Pepkowitz (*Anal. Chem.*, 1955, **27** [2], 245–248).—The continuous oxygen-detector and analyser described and illustrated is constructed by making the following additions to and modifications of the G.E.C. photo-electric dew-point recorder: multi-circuit switch, gas-inlet system, and circuits for minor-heater and platinum-catalyst furnace with pyrometer; the circuit modifications enable the instrument to act either as a dew-point recorder or as a continuous oxygen-detector. The temp. of the heated mirror is set to the dew-point corresponding to the specified concn. of O in the gas when converted into H_2O. At all H_2O concn. less than this value, the mirror remains clear and the alarm silent; otherwise frost forms and the alarm system is actuated. The approx. total O content of the gas (including H_2O) is obtained by determining the normal dew-point of the gas and the dew-point of the gas plus added hydrogen. The oxygen value is the difference between these two values expressed as vol.-per cent. of H_2O divided by two. The response of the instrument is rapid, the sensitivity is about 0·0005 per cent. by vol. of O, with a mean deviation for precision of $\pm$ 0·0001 per cent. by vol. Since the flow of H is $\simeq$ 5 ml per min., whilst that of the test-gas is $\simeq$ 2800 ml per min., the oxygen contribution of the added H is negligible. The apparatus can be used for determining oxygen in N, He, H, CO_2 and the rare gases. Its normal use is in liquid-metal heat-transfer systems where concn. of O must be kept below 0·001 per cent. v/v.
W. J. BAKER

1976. **The Gooderham - G.L.C.C. soap-film gas-analysis apparatus.** G. R. Scholes (*Gas J.*, 1955, **281**, 319–322).—The operation of the apparatus is described in detail. The gas passes through a series of soap-film meters in which a soap film is carried upwards by the gas until the meter is by-passed; the position of the film is then read off on the graduated meter. Between the meters, one constituent of the gas is removed by the usual means; the residual gas in the last meter is N. Town's gas experience at Oldham gas works indicates an accuracy such that calculated calorific values are within $\pm$ 3 B.Th.U. of the determined values. Regular changing of solutions and catalysts is necessary.
J. G. KING

1977. **Potentialities of the sonic gas analyser.** A. E. Martin (*Ind. Chem.*, 1955, **31**, 60–62).—The principle and theory, and a practical form of a sonic gas analyser, are described; its applications in gas analysis are indicated.
D. R. PECK

1978. **Improved interferometric means for measuring the composition of gases.** Z. Tuzi (Brit. Pat. 725,748, Date Appl. 28.5.53).—A modified interferometric method for measuring the composition of gases is described and illustrated. Two parallel beams from the same light source are passed, respectively, through standard and test gas chambers, reflected through 180° and returned through the same chambers and finally recombined for optical evolution of fringe displacement caused by variations of refraction in the test chamber. The methods for producing and recombining the two beams and of reflecting each through 180° are illustrated.
D. BAILEY

1979. **Modified nitrometer.** B. D. Holt (*Anal. Chem.*, 1955, **27** [2], 334).—In the nitrometer assembly described and illustrated the burette (100 ml) and compensating tube are both enclosed in a water-jacket and are connected only by a mercury manometer, each tube having its own levelling bulb. After standardisation of the apparatus (with NO liberated from a known wt. of KNO_3), only the levelling bulb of the burette need be moved during each analysis. The 200-ml reaction bulb enables a lighter load of mercury to be handled. The principle involved in the gas measurements is the same as for the conventional Du Pont nitrometer. A vol. of dry air in the compensating tube is compressed to the vol. it would have at a standard temp. and pressure, and the pressure of the NO in the burette is then equated to that in the compensating tube (as indicated by the manometer) so that its vol. (V) is equal to that at the standard

conditions. Then, the percentage of nitrogen compound = (100 V × mol. wt.)/(22,400 × sample wt.).
W. J. BAKER

1980. **Laboratory gas-flow control device.** R. A. Hall (*J. Sci. Instrum.*, 1955, **32** [3], 116–117).—The device described compensates for fluctuations in supply and apparatus gas pressures and maintains a constant pressure-difference across the flow-meter capillary without the necessity of venting gas out of the apparatus. G. SKIRROW

1981. **A fifty-stage apparatus for distillation at very low pressures.** B. J. Mair, A. J. Pignocco and F. D. Rossini (*Anal. Chem.*, 1955, **27** [2], 190–194).—The design, assembly, operation and testing of a 50-stage apparatus for continuous distillation at pressures of 0·01 to 0·1 mm of mercury are described fully. Results for a distillation of $\simeq$ 2·7 litres of a commercial mixture of normal paraffins (C_{18} to C_{32}) and of a concentrate of normal paraffins (C_{17} to C_{25}) from petroleum are reported. Both distillations were conducted at v.p. $\simeq$ 0·03 mm of mercury, a throughput of 80 ml of liquid per hr., and a reflux ratio of 55 : 1, with a total distilling time of 1900 hr. Continuous distillation for $\simeq$ 6 hr. is necessary to obtain a steady state. Experience indicates that a similar 100-stage apparatus would be practical. W. J. BAKER

1982. **Vapour-recirculating equilibrium still.** H. Hipkin and H. S. Myers (*Ind. Eng. Chem.*, 1954, **46** [12], 2524–2528).—In the improved vapour-recirculating still described and illustrated, adiabatic conditions are maintained by a separate vapour-jacket into which are sealed two 250-W heaters, one for direct heating and the other operating intermittently from a differential temp. controller. The use of Teflon valves eliminates stopcocks and grease; temp. can be held to ± 0·1° C, whilst equilibrium is rapidly attained and operation is almost entirely automatic. The still has negligible entrainment and low pressure-drop. Vapour-liquid equilibrium data obtained by operating this still with the systems heptane - toluene and methanol - carbon tetrachloride are in good agreement with published data obtained previously for the same systems from other types of still.
W. J. BAKER

1983. **Modified ASTM distillation apparatus for 10-ml samples.** A. E. Fruhwirth (*Erdöl u. Kohle*, 1955, **8**, 34–35).—The dimensions of a miniature distillation apparatus are given. The results compare closely with those of the standard 100-ml distillation apparatus (Amer. Soc. Testing Materials D-86-52) except for a slightly lower end temp. and a smaller yield of distillate (*Cf.* Wiberley and Siegfriedt, *Petroleum Engr*, 1953, **25** [9], 12).
A. R. PEARSON

1984. **Vertical umbrella-type agitator to promote smooth boiling in vacuum distillation.** A. Rose and W. W. Sanders (*Anal. Chem.*, 1955, **27** [2], 331–332).—The device consists of a vertical glass rod along which are sealed at intervals about four or five inverted-cup glass baffles, each with several holes drilled through it. The upper end of the shaft is a short piece of glass tube housing a small soft-iron core, whilst a stainless-steel bumper spring (movable) is attached to the lower end of the shaft. When the external magnet around the core is energised the stirrer moves up, and drops again when the circuit is broken. A rate of 50 to 150 strokes per min. ensures smooth steady boiling *in vacuo* at high rates of vaporisation (2 to 3 litres per hr.) and for time periods up to 170 hr. The stirrer is particularly useful for such liquids as methyl oleate and similar esters, hexoic acid and acids or alcohols having an even number of C atoms from C_6 to C_{18}.
W. J. BAKER

1985. **New serviceable laboratory centrifuges. (Preliminary communication.)** E. Wiedemann (*Helv. Chim. Acta*, 1955, **38** [1], 37–46).—The new range of "Syrogyro" laboratory centrifuges is described. These centrifuges have closable rotors of 200, 500 and 1000 ml capacity, which hold eight vessels of 25 ml, 62·5 ml and 125 ml capacity, respectively, and a regulatable electric drive, which can give a speed of 20,000 r.p.m., equivalent to a gravity value of 40,000. The speed is reached in only 2 to 3 min. and a special electric braking device enables the rotor to be brought quickly to rest (4 to 5 min.). A special system of air cooling is incorporated, which prevents heating up of the substances being centrifuged. The vessels used for centrifuging are of synthetic resin or V4A steel, which enables them to be used for handling substances of all kinds. Laboratory separations can be effected rapidly and efficiently. The use of the machine for quantitative sedimentation of chloroplastin and of milk casein from fresh milk is described in detail. H. L. WHITEHEAD

1986. **Viscometer for solutions of high polymers.** C. D. Keeling and M. Dole (*J. Polymer Sci.*, 1954, **14** [73], 105–111).—A viscometer for solutions of high polymers is described having the features: (*i*) the lower level of the capillary viscometer is determined by an overflow into an auxiliary reservoir; (*ii*) the solutions are prepared, the insoluble gel is filtered and dilutions are carried out in the auxiliary reservoir; (*iii*) because the viscometer is a closed system it can be rotated through 360° within the constant-temp. bath, automatically filling the upper reservoir and allowing repetitive measurements to be carried out. As constructed, the viscometer can be used only for solutions of low surface tension.
PAINT RESEARCH STATION ABSTR.

1987. **Capillary viscometer with continuously varying pressure head.** S. H. Maron, I. M. Krieger and A. W. Sisko (*J. Appl. Phys.*, 1954, **25** [8], 971–976).—A capillary viscometer has been designed to study the flow behaviour of non-Newtonian fluids. The instrument covers continuously a wide shearing-stress range in a single determination. A falling-mercury column forces the sample through the capillary. Measurements of the column height as a function of time give both pressure drop and flow rate. Provision is also made for the use of driving fluids less dense than Hg for measurements at lower shearing stresses. The density of the sample need not be determined. Methods and illustrations are given of the application of the instrument to the determination of the absolute viscosities of Newtonian liquids and the flow curves of non-Newtonian fluids.
PAINT RESEARCH STATION ABSTR.

1988. **A Pirani gauge circuit.** C. N. W. Litting (*J. Sci. Instrum.*, 1955, **32** [3], 91–92).—The circuit diagrams described include a germanium rectifier used in conjunction with a direct-reading constant-temp. Pirani gauge. The calibration curve is almost linear. G. SKIRROW

1989. **Differential dilatometers.** A. Masenauer (*Sklář a keramik*, 1954, **4** [2], 36–41; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 41,775).—A differential dilatometer for determining the coefficient of expansion of various materials is described.
E. HAYES

1990. **Extensometer for testing putty and similar materials.** G. E. Bessey and K. A. Lammiman (*J. Oil Col. Chem. Ass.*, 1955, **38** [2], 92–98).—A detailed description is given of the extensometer designed specifically for testing the tensile strength and extension at break of linseed-oil putty. The design is based on a research instrument that has already been described (*Bull. Brit. Soc. Rheol.*, 1952, [35], 7; *J. Oil Col. Chem. Ass.*, 1954, **37**, 562). The instrument has a fixed rate of extension and may be used to indicate the load and the extension at break, which can be read off from a dial or from a drum chart; it may also be used to record the load - extension curve. The results are repeatable to ± 20 g for max. load and to about ± 1 cm for extension at break.
S.C.I. ABSTR.

1991. **Self-adjusting and dispensing micro-pipette.** B. J. Grunbaum and P. L. Kirk (*Anal. Chem.*, 1955, **27** [2], 333).—The pipette is designed to deliver successive aliquots of sample, or const. vol. of wash liquid, with effective and controlled rinsing. It is in effect a measuring tube with five capillaries on both ends to permit adjustment of the vol. after filling from either end. On the top is sealed a reservoir bulb with tapered opening that fits into a syringe-pipette control or a rubber tube. The capillary is divided into two equal vol. by a calibration mark at its centre. The method of using the pipette for measuring organic solvents, viscous solutions, or similar liquids that are difficult to remove by conventional rinsing is described.
W. J. BAKER

1992. **Tobacco investigations. A new automatic smoking apparatus.** M. Staub and H. Furrer (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [6], 473–476).—A description is given of a new electronically controlled and fully automatic apparatus that smokes cigarettes.
R. J. MAGEE

1993. **Apparatus for measuring the elastic properties of gels.** L. J. Hastewell and R. Roscoe (*J. Sci. Instrum.*, 1955, **32** [3], 96–99).—A cylindrical float is placed co-axially in a vessel containing the suspension. A rotating movement is given to the float by directing jets of air on to two vanes that are attached to a spindle that is co-axial with the float. The subsequent oscillations of the float are followed by a beam of light directed on to a mirror that is attached to the spindle and brought to a focus on a drum camera. After calibration with a liquid of known viscosity and the same density as that of the gel, the dynamic rigidity and dynamic viscosity can be calculated from the frequency and decrement of the oscillations.
G. SKIRROW

See also Abstracts 1723, 1725, 1850.

Optical

1994. **Simple extrusion press for preparation of specimens of optimum thickness for X-ray diffraction powder photographs.** M. Kossenberg (*J. Sci. Instrum.*, 1955, **32** [3], 117–118).—The specimen is formed by compression in a capillary holder that has a polished bore. During compression, the lower end of the capillary is maintained against a block. After compression, the block is removed and, by means of a special punch, the specimen is pressed out about 3 mm. The specimen holder fits directly into the rotating support in a powder camera.
G. SKIRROW

1995. **General investigation of photo-electric spectrophotometers.** J. A. A. Ketelaar, J. Fahrenfort, C. Haas and G. A. Brinkman (*Chem. Weekbl.*, 1955, **51** [12], 211–219).—The reliability of optical density determinations carried out with a number of spectrophotometers has been investigated. Seventy-eight spectrophotometers were used: 41 Beckman u.v. spectrophotometers, 29 Unicam instruments, 6 Beckman instruments with tungsten lamp, a Zeiss - Opton and a Cary recording spectrophotometer. For 15 of the instruments, observations were carried out by two observers, with $K_2Cr_2O_7$ as test substance. Analysis of the results shows that duplicate estimates give no impression of the accuracy of optical density measurements. A particular instrument may give extinction readings that are systematically too high or too low. The standard deviation for this effect is ± 1·3 per cent. The differences between cells have a standard deviation of ± 1·1 per cent. Optical density measurements with different instruments will therefore have a standard deviation of ± 1·7 per cent., whilst in the determination of concentrations with one instrument there is a standard deviation of ± 1·6 per cent.
A. J. MEE

1996. **Spectrometric apparatus.** Zeiss - Opton Optische Werke Oberkochen G.m.b.H., trading as Carl Zeiss (Brit. Pat. 725,508, Date Appl. 5.8.53; Ger. Pat. 18.8.52).—This is an addition to B.P. 667,890, which relates to a spectrometer, equipped with a rotating dispersion element (refracting prism) and a stationary wavelength scale, in which an image of the entrance slit is used as a reading index for the scale. The rotatable dispersion element is replaced by a stationary element and a principal rotatable mirror which scans the spectrum across the exit slit. The entrance slit is focused on the wavelength scale by light reflected by a reflecting plane (firmly attached to the dispersing element) and by an accessory mirror which is fixed to the principal mirror and rotates with it.
J. M. JACOBS

1997. **The design and construction of a 22-foot direct-reading optical spectrometer.** T. Lee (*Applied Spectroscopy*, 1954, **8** [4], 174–207).—The instrument incorporates a 5-in. concave grating ruled with 15,000 lines per in. in an Eagle mounting. Dispersion is 2·6 A per mm in the first order and 5 feet of spectra are available at each setting. Provision is made for multiplier photocells to be fixed to receive certain lines or for a single cell to scan the spectrum. A ratio recorder measures the ratios of either instantaneous or integrated voltages. Circuits are given which are capable of a precision of 0·088 per cent. in the measurement of the intensity ratio of two argon lines from a discharge tube.
P. T. BEALE

1998. **Segregation in cast aluminium alloy spectrographic electrodes.** W. E. Mew, F. H. Smith and J. Wood (*Metallurgia*, 1955, **51** [303], 45–51).—Non-uniformity has been confirmed in several types of

flat cast aluminium alloy electrodes by examination with a direct-reading spectrograph. The effects of segregation can be largely overcome by selective sparking in predetermined locations. Similar remarks apply to cast pencils, although longitudinal variation has not proved excessive, but an improvement of about 25 per cent. in reproducibility is indicated with wrought electrodes. The contribution to discharge intensity is also shown to be non-uniform over the area covered by the spark; the centre and, in pencil electrodes, the edge, make a major contribution. J. O. LAY

1999. **A drum attachment to a travelling microscope for direct wavelength reading of spectral lines.** S. A. Clarke (*Spectrochim. Acta*, 1955, **7** [1] 64).—By fixing a pen to the pointer on the microscope stage, a helical expanded distance scale can be traced on a rotating drum geared to the thread of the travelling microscope. The helix is calibrated for λ by recording microscope scale-readings of known lines in reference spectra (photographed in juxtaposition), and constructing therefrom a table of scale readings corresponding to 10-A intervals over the range 2160 to 6000 A. This table can be subdivided proportionately into smaller units. The procedure for obtaining a direct reading of wavelength from the drum is described. The mean difference between indicated and true λ of single readings on many spectral lines ranges from $\simeq$ 0·25 to 0·90 A according to the spectrograph used. Errors are unidirectional and progressive in amount. W. J. BAKER

2000. **Compact monochromator employing rhombic constant-deviation prisms.** J. W. Gates and S. P. Middleton (*J. Sci. Instrum.*, 1955, **32** [3], 113–115).—Details are given of the design of a monochromator that comprises two 60° rhombic prisms, arranged to give a total deviation of 240°. The entrance and exit apertures are so arranged that they can be rapidly replaced by others of different size. G. SKIRROW

2001. **Modified viewing-chamber windows for electron microscopes.** G. C. Claver, jun. (*J. Opt. Soc. Amer.*, 1955, **45** [2], 132).—A laminated window is described, suitable for use with R.C.A. electron microscopes of Series 2, in which the components are of such dimensions as to leave a recess to hold the normal sealing gasket. With windows of this type, it is almost impossible for the gasket to be drawn in under vacuum. B. S. COOPER

See also Abstract 1886.

Thermal

2002. **Glass surface temperature pyrometer.** J. R. Beattie (*J. Soc. Glass Tech.*, 1954, **38** [184], T 457–T 469).—In an instrument for the range 150° to 550° C, the radiation 4·5 to 8 μ (in which region the glass functions as a "black body") is filtered out (by means of a dehydrohalogenated film of polyvinyl chloride, with a fumed film of MgO on its surface) and directed on a Schwarz-type FT17 vacuum thermopile. Cooling water, from a thermostat at 22° C, is circulated through the housing of the instrument. A hollow cube of cast iron within a furnace whose temp. is measured with a thermocouple is used for calibration purposes. Corrections for (*i*) variation of the thickness of the glass from $\frac{1}{4}$ to $\frac{1}{16}$ in. varied from + 3° to − 11° C, (*ii*) variation of ambient temp. of the instrument housing from 16° to 28° C varied from +2·5° to −1·5° C and (*iii*) variation of the distance between the surface and the pyrometer of 17 to 21 in. were negligible. The calculation of the total energy emitted by glass having a given surface temp. and temp. gradient is given. J. A. SUGDEN

2003. **Oxy-hydrogen burner for determination of sulphur in drip oils.** L. Granatelli (*Anal. Chem.*, 1955, **27** [2], 266–269).—A method, in which the sample is burned in the oxy-hydrogen burner of the Beckman flame photometer, is described for the determination of S in drip oil. The products of combustion are absorbed in a soln. of 3 per cent. H_2O_2 and the S is determined gravimetrically as $BaSO_4$. Analysis of samples containing 0·006 to 0·112 per cent. of S showed standard deviations of $\pm$ 0·001 to $\pm$ 0·004 per cent. Improvement was obtained by modification of the apparatus, a sample containing 0·058 per cent. of S giving a standard deviation of 0·001 instead of 0·004 as formerly. The suitability of the method to the determination of total S, and to the determination of halogens in various petroleum products, is discussed. G. P. COOK

2004. **Adiabatic calorimeter for measuring the specific heats of liquids in the range 0° to 100° C.** L. W. Phipps (*J. Sci. Instrum.*, 1955, **32** [3], 109–113).—The calorimeter suitable for 125 ml of liquid is maintained in an electrically heated annular jacket and is equipped with a heater that gives wide dispersal of heat without local overheating. Agitation of the fluid is effected by oscillation of the calorimeter about its vertical axis. An accuracy of $\pm$ 0·5 per cent. is given with temp. rises of about 3° C. G. SKIRROW

Electrical

2005. **Photo-electric polarimeter attachment.** H. Rudolph (*J. Opt. Soc. Amer.*, 1955, **45** [1], 50–59).—A photo-electric attachment, suitable for use with a conventional type of polarimeter, is described and the performance assessed. For monochromatic sodium-yellow or mercury-green illumination, the precision of the photo-electric and visual methods is similar, provided that large apertures and high light transmission of samples can be maintained and that the visual observations are made under conditions of dark-adaptation of the eye. Under other experimental conditions, the photo-electric method can give higher precision than the visual method. B. S. COOPER

2006. **Studies on electrolytic analysis with an automatically controlled potential.** S. Oka, G. Muto and S. Nagatsuka (*Japan Analyst*, 1953, **2** [3], 198–201).—An apparatus for electrolytic analysis has the potential automatically controlled by means of a multivibrator. A rectangular wave, which is generated at $\simeq$ 2000 cycles by a push-pull valve, is amplified, rectified with a selenium rectifier and supplied to the electrolytic cell. A constant potential is maintained by regulating the voltage of the rectangular wave with a potentiometer and a voltage-regulating valve. Electrolytic determination of Cu in brass in HCl soln. containing hydroxylamine-HCl (at − 0·36 V *vs.* the S.C.E.) in the presence of Sn was carried out with this apparatus. K. SAITO

2007. **Apparatus difficulties and sources of error in paper electrophoresis. Separation of organic acids.** H. Berbalk (*Monatsh. Chem.*, 1954, **85**, 1314–1319).—The conditions of temperature, moisture in the strip, diffusion, constancy of composition of the solution, migration of the solution, and the endosmotic effect are investigated since they are important factors in exact electrophoretic measurement and analysis. A description of the apparatus employed is given and, with the stated conditions of experiment, carboxylic acids, sulphonic acids and strongly acid phenols have been separated.
R. J. MAGEE

2008. **Recording integrating photo-electric and radioactive scanner for paper electrophoresis and chromatography.** E. L. Durrum and S. R. Gilford (*Rev. Sci. Instrum.*, 1955, **26** [1], 51–56).—Paper chromatograms are scanned automatically by an instrument which records directly optical density or radioactivity. For optical scanning, a narrow-band light source is obtained by interference filters, or monochromatic light is obtained from a spectrometer monochromator. For radioactive scanning, a Geiger - Müller tube replaces the photocell in the optical unit. For low activities, optical scanning of autoradiographs is satisfactory. G. SKIRROW

2009. **Studies on a.c. polarography. II. Fundamental circuit and some experimental results.** M. Senda, M. Okuda and I. Tachi (*Bull. Chem. Soc. Japan*, 1955, **28** [1], 31–36).—A polarograph is described in which a small alternating potential is superimposed upon the direct applied potential and the a.c. recorded automatically on photographic paper. The amplitude of the alternating voltage may be constant or varied. A short discussion of the experimental characteristics of the circuit is given. H. F. W. KIRKPATRICK

2010. **Studies on a.c. polarography. III. Reversible wave.** M. Okuda and I. Tachi (*Bull. Chem. Soc. Japan.*, 1955, **28** [1], 37–41).—Experimental results with Cd^{++} and Tl^{+} using the polarograph described earlier (see *Anal. Abstr.*, 1955, **2**, 2009) are correlated with theoretical considerations of the a.c. polarography of reversible reductions at the dropping-mercury electrode.
H. F. W. KIRKPATRICK

2011. **A stationary mercury electrode for polarographic micro-analysis with flowing electrolyte and its importance for the detection of trace elements in blood.** G. Kahle and E. Reif (*Biochem. Z.*, 1955, **326** [4], 305–310).—The design and properties are given for a stationary mercury electrode that is suitable for the determination of elements such as Cu, Cd and Zn in a small volume of blood.
G. W. CAMBRIDGE

2012. **Effect of magnetic stirrers on pH measurements.** G. E. Livingston (*Chemist Analyst*, 1954, **43** [4], 106).—Errors may be introduced into the electrometric determination of pH by the use of magnetic stirrers. At low speeds (0·3 max.) low results (from 0 to 0·20 units, according to the pH range) were obtained; at pH 7·0, the observed pH was lower by 0·03 units for each 10 per cent. increase in speed; at high speeds, considerable fluctuations were observed in the electrometer. The effect appears to be due to the rotating field of the stirrer bar and it may be advisable to take pH readings only with the stirrer switched off.
G. B. THACKRAY

2013. **Appliance for measuring the pH value of biological fluids.** R. C. Noyes (Brit. Pat. 725, 586, Date Appl. 18.3.53).—A glass electrode for measuring the pH of biological fluids is described and illustrated. The electrode, in which a ratio of 1:100 between the inner volume of the electrode capillaries and liquid volume surrounding them is obtained, is designed to determine the pH of < 100 mg of liquid with an accuracy of at least 0·01 pH unit. The apparatus also comprises an auxiliary appliance for the glass electrode adapted to collect blood from an incision in the skin and at the same time to exclude air. D. BAILEY

2014. **An RF linear decelerator mass spectrometer.** W. Donner (*Applied Spectroscopy*, 1954, **8** [4], 157–162).—The ion beam is given a high velocity in a linear accelerator of the Sloan type and subsequently passes through a series of holes in plates, which are connected to an RF sweep oscillator (3 to 10 Mc), and which cause preferential deceleration of the resonant particles by an amount equal to the root mean square value of the RF voltage at each gap, the other ions being correspondingly accelerated. The resultant beam is separated in an electrostatic field and the preferred component of lowest energy is collected in a Faraday bucket. Advantages are compactness, with resolution to mass 100, and elimination of troubles associated with magnetic field in the region of source and collector.
P. T. BEALE

2015. **A two-stage magnetic analyser for isotopic ratio determinations of 10^4 to 1 or greater.** F. A. White and T. L. Collins (*Applied Spectroscopy*, 1954, **8** [4], 169–173).—Constructional details are given of an arrangement of two tandem magnets which eliminates most of the problems associated with the elastic scattering of positive ions and so permits the detection of very small peaks when they are adjacent to very large ones. Mass spectra of K, Rb, W and Ba illustrate the advantages of the two-stage analyser over the single-stage instrument.
P. T. BEALE

2016. **Technique for continuous intensity standardisation in quantitative analysis by nuclear magnetic absorption.** R. H. Elsken and T. M. Shaw (*Anal. Chem.*, 1955, **27** [2], 290–292).—In the nuclear-resonance spectrometer circuit described and illustrated provision is made for continuous comparison (by servo-system) of the absorption signal with an electronic standard of reference produced in a Watkins - Pound calibrator circuit. The system is maintained in continuous zero-balance, and the specimen signal, calibrator-input signal and the resulting artificial absorption signal are all detected as sinusoidal variations; 20 min. after the start, readings are consistent to within $\pm$ 0·3 per cent. The procedure, which is claimed to give greater accuracy of line intensity, can be used for the determination of water in hygroscopic solids (by proton resonance absorption), for relaxation-time measurements, and where a change in spectrometer radio-frequency energy level in the specimen coil is required while holding the system-gain constant.
W. J. BAKER

2017. **Method and apparatus for indicating and controlling moisture content [of granular material].** National Coal Board (Inventor: D. J. Millard). Brit. Pat. 721,223, Date Appl. 30.1.53.—The granulated material, comminuted coal, sand or gravel, is allowed to flow through or near a condenser altering its capacity and unbalancing a high-frequency (> 10 megacycles) bridge. The unbalance of the bridge is measured by a valve voltmeter. As such samples are usually conductive, alternative circuits are described. For spot samples, a variable resistance across the standard capacity is adjusted to balance the conductivity of the sample; for moving continuous sampling a reference current from the oscillator is fed to the detector of the valve voltmeter, so that the indicator responds only to the reactive unbalance of the bridge. A form of fringe-field capacitor suitable for moving streams of coal is described.

E. J. H. BIRCH

See also Abstracts 1796, 1798.

ERRATUM.—April (1955) issue, abstract 1011, line 11.
Insert "(apparent to unknown)" after "potency ratio."

Notice

The Editor will be pleased to supply on request addresses of authors of papers, if these are available, or of the journals from which abstracts have been prepared.

Translations

The following papers of interest to analytical chemists have been translated into English. Copies of these papers can be obtained from Consultants Bureau, 152, West 42nd Street, New York 18, N.Y., U.S.A. Each translation costs $7.50 and orders should state title, author(s) and English page number. The English page number is given in parentheses after the Russian page number.

These translations can also be seen in the library of the Chemical Society, Burlington House, London, W.1.

J. Anal. Chem., U.S.S.R.—

Oscillographic polarography—Ya. P. Gokhstein and Yu. A. Surkov, 1954, **9**, 319 (355).

A flame photo-electric method for the determination of calcium in solution—D. I. Ivanov, 1954, **9**, 344 (383).

Quantitative colorimetric determination of aliphatic alcohols in the ultra-violet—S. A. Shchukarev, S. N. Andreev and I. A. Ostrovskaya, 1954, **9**, 354 (393).

Quantitative micro-determination of carbon, in particular in tantalum carbide—V. I. Smirnova and B. F. Ormont, 1954, **9**, 359 (399).

An apparatus for the quantitative absorption of gases—B. F. Ormont and V. I. Smirnova, 1954, **9**, 364 (405).

Polarographic determination of nitrates and nitrites in salts and brines used for meat pickling—R. Pletikha and E. Krzhishova, 1954, **9**, 366 (407).

A study of the errors involved in the chemical analysis of steels and casts—A. B. Shaevich, 1954, **9**, 373 (415).

Refractometric analysis of ternary systems—B. V. Joffe and A. G. Morachevsky, 1955, **10**, 3 (1).

X-ray spectrographic determination of hafnium in zirconium minerals and concentrates—E. E. Vainshtein, I. D. Shevalevsky and I. F. Shtauberg, 1955, **10**, 14 (11).

Quantitative spectrographic analysis of hafnium in zirconium—G. Kryuger, R. R. Shvangiradze and T. A. Mozgovaya, 1955, **10**, 20 (15).

Colorimetric determination of vanadium by means of a catalytic reaction—V. A. Nazarenko and E. A. Biryuk, 1955, **10**, 28 (21).

Organic co-precipitants. II. Co-precipitation of tungsten with organic co-precipitants—V. I. Kuznetsov, M. N. Obozhin and E. S. Palshin, 1955, **10**, 32 (25).

Potentiometric titration using the catalytic decomposition of hydrogen peroxide on a platinum electrode—I. E. Flis, 1955, **10**, 38 (31).

Potentiometric determination of Cr_2O_7'' and strong acids when both are present simultaneously—I. E. Flis and Zh. L. Verl, 1955, **10**, 44 (37).

Functional analytical groups for iron and bismuth—N. M. Turkevich, 1955, **10**, 47 (41).

Micro-determination of carbon in metals—P. A. Kryukov and E. V. Rengarten, 1955, **10**, 51 (45).

Precipitation of quadrivalent vanadium with 5:7-dibromo- and 5:7-di-iodo-8-hydroxyquinoline—I. P. Alimarin and V. G. Kryukov, 1955, **10**, 56 (49).

Diphenylcarbazone as a reagent for the colorimetric determination of germanium—G. S. Desmuk, 1955, **10**, 61 (53).

A specific reaction for formaldehyde—G. Vanag and E. Vanag, 1955, **10**, 63 (55).

Characteristic chromatographic reactions for glucose and acetone—F. M. Shemyakin and O. S. Lobakhina, 1955, **10**, 65 (57).

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångstrom unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
critical	crit.
crystalline, crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c.p.s.
decompos -ing, -ition	(decomp.)
density	ρ
density, relative	d or wt. per ml
derivative	deriv.
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
electron-volt	eV
equivalent	equiv.
experiment	expt.
foot, feet	ft.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
horse-power	h.p.
hour	hr.
hydrogen ion concentration	$[H^{\cdot}]$
hydrogen ion exponent	pH
inch	in.
infra-red	i.r.
insoluble	insol.
kilogram	kg
kilovolt	kV
kilowatt	kW
maxim -um, -a	max.
melting-point	m.p.
microcurie	μC
microgram	μg
microlitre	μl
micron	μ
milliampere	mA
millicurie	mC
milligram	mg
millilitre	ml
millimetre	mm
millimicron	mμ
millivolt	mV
minimum	min.
minute (time)	min.
molar (concentration)	M
molecul -e, -ar	mol.
normal (concentration)	N
number	no.
observed	(obs.)
ounce	oz
part	pt.
patent	pat.
parts per million	p.p.m.
per cent. wt. in wt.	per cent. w/w
per cent. wt. in vol.	per cent. w/v
per cent. vol. in vol.	per cent. v/v
potential difference	p.d.
pound	lb
precipitate	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_{λ}
relative humidity	R.H.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_{\lambda}^{t}$
square centimetre	sq. cm
standard temperature and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, $Cu^{\cdot\cdot}$, $Al^{\cdot\cdot\cdot}$, Cl', SO_4''. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe^{III} and cuprous copper Cu^{I}.

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